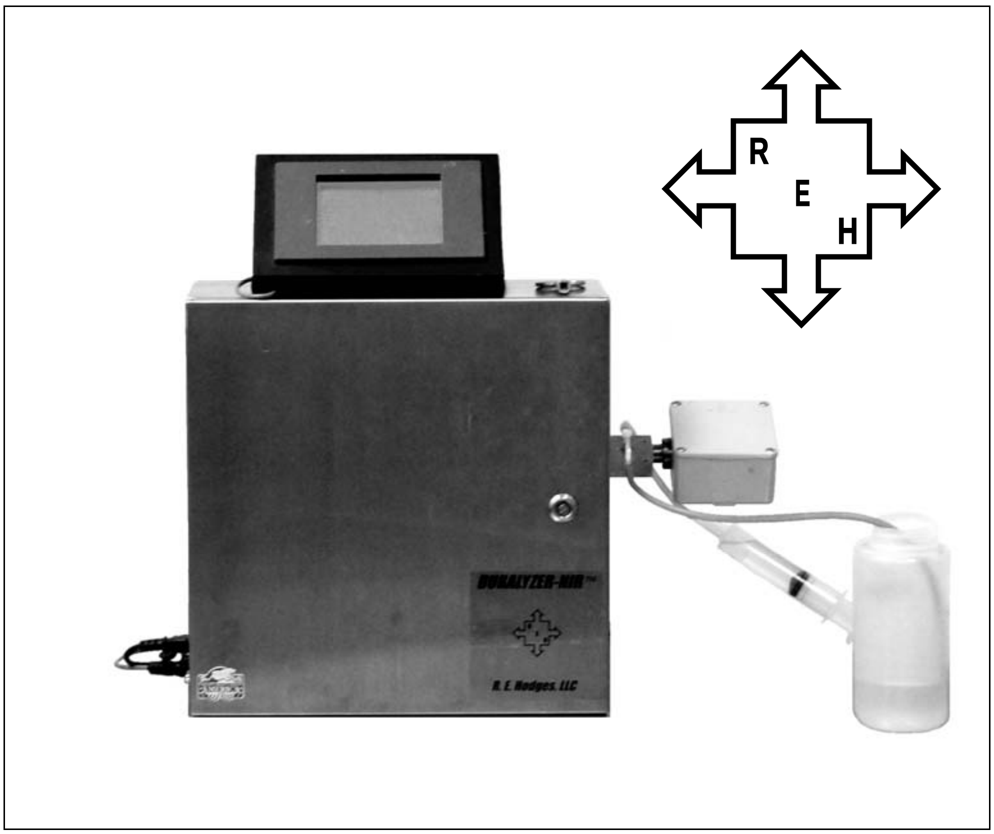
DURALYZER-NIR ™

Laboratory Liquor Analyzer

March 2009

User Manual



R. E. Hodges, LLC

Pioneering Advanced Measurement Solutions
for the Process Industries

Company Profile

R. E. Hodges, LLC (hereafter referred to as REH, LLC) was formed in 2001 as a manufacturer of online and laboratory spectroscopic based measurement solutions for the process industries. The equipment and instrumentation developed by REH, LLC is specifically designed to be easily incorporated into a control strategy.

In general, spectroscopic based measurements hold the key to unlocking the majority of difficult measurement applications in the process industries. REH, LLC was created to replace or reduce traditional laboratory based testing for quality control by implementing real time online measurements coupled with practical control strategies. Unlike traditional spectrometer manufacturers, REH, LLC provides turnkey sampling and measurement solutions that are tailored to suit the specific application. In addition to accuracy and reliability, our analyzers are characterized by minimal installation and continuing maintenance requirements.

At REH, LLC, we take great pride in the innovation and quality of our products and the continuing service and support we provide for each product. We are unique in our ability to work with the customer to develop a customized online or laboratory measurement solution. Our overall goal is to continually improve and expand our product line, capabilities, and service to meet the evolving challenges of current and future customers. It is our firm belief that the customer's success translates into our success.

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About This Manual

This manual provides information necessary for proper operation and care of the ***DURALYZER-NIR™*** laboratory liquor analyzer. The subject matter is divided into four main headings.

1. Introduction and Specifications
2. Principle of Operation
3. Installation
4. Operation and Maintenance

The manual is more than a simple set of instructions. Specific topics covered include the following:

- Comparison between the ***DURALYZER-NIR™*** and other current solutions (Chapter 1).
- General specifications (Chapter 1)
- NIR spectroscopy and its implementation (Chapter 2)
- Installation guidelines and procedure (Chapter 3)
- Startup and normal operation (Chapter 4)
- Customizing the analyzer settings (Chapter 4)
- MODBUS configuration (Chapter 4)
- Calibration model tuning (Chapter 4)
- Maintenance requirements and procedure (Chapter 4)

Information contained in the main body of the manual is further detailed in the appendices.

Careful attention has been paid to accuracy of the contents of this manual. R. E. Hodges welcomes any recommendations, suggestions or corrections that would serve to improve the quality and utility of this literature.

Disclaimer

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1 Introduction and Specifications

1.1 Introduction

The ***DURALYZER-NIR™*** laboratory liquor analyzer has been engineered to provide fast, reliable and accurate measurements while avoiding the negative issues associated with manual testing and laboratory autotitrators. The laboratory liquor liquid analyzer shares the near-infrared (NIR) technology utilized by our online liquor analyzers and is specifically designed to function in the somewhat harsh laboratory environments in the pulping and recovery areas. Maintenance requirements have been held to a minimum, consisting of periodic acid cleaning of the sample cuvette and annual light source replacement. The ***DURALYZER-NIR™*** laboratory liquor analyzer is shown in Figure 1-1.

Figure 1-1. ***DURALYZER-NIR™*** laboratory liquor analyzer.

Effective alkali (EA), active alkali (AA), total titratable alkali (TTA), total dissolved solids (TDS), and total dissolved dead load (TDD) are computed for white liquor, green liquor, and weak wash. Additionally, Na_2SO_4 is measured for green liquor to allow for

determination of reduction efficiency (RE). For black liquors, analysis consists of residual effective alkali (REA) and TDS. As an option, residual active alkali (RAA) and lignin measurements can be provided for black liquor samples.

1.2 *DURALYZER-NIR™* vs. Current Solutions

Manual (laboratory) liquor testing is performed routinely for quality control purposes. For pulp mills utilizing online liquor analyzers, periodic testing is still necessary in order to validate the online measurements. Frequently, lab tests are the only source of measurements for process control decisions. The rather tedious and cumbersome nature of the standard "ABC" testing procedure for process liquors does not lend itself to rapid manual testing. Unfortunately, it does lend itself to induced errors and biases. As a result, liquor testing frequency is low (once or twice per operator shift) and the ability to reduce or correct process variations and disturbances is severely hampered. A brief comparison between the standard "ABC" titration test, laboratory autotitrators, and the ***DURALYZER-NIR™*** laboratory liquor analyzer is shown in Table 1-1.

Table 1-1. ***DURALYZER-NIR™*** vs. Current Solutions.

Characteristic	STD "ABC" Titration	Autotitrator	*DURALYZER-NIR™*
Available Measurements	3 (EA, AA, TTA)	3 (EA, AA, TTA)	7 (EA, AA, TTA, TDS, TDD, Lignin, Na_2SO_4)
Measurement Technique	Direct: volumetric analysis	Inferred: inflection pt. method based on pH titration curve (SCAN method)	Inferred: regression model relating NIR spectral signature to chemical composition
Measurement Accuracy Factors	Potential volumetric errors	Potential volumetric errors, difficulties in pinpointing inflection pts. (especially for AA)	no volumetric errors
Analysis Speed	Slow: minutes to 10's of minutes	Slow: minutes to 10's of minutes	Fast: 20 to 60 seconds
Maintenance	Low: maintain glassware and chemicals	Moderate: acid replacement, pH probe calibration, occasional lab validation	Low: annual light source replacement, occasional lab validation

The standard "ABC" titration test requires multiple chemicals and lab equipment to implement. Titration chemicals such as certified hydrochloric acid solution, formaldehyde, barium chloride and various color indicators can be inconvenient (in terms of cost and space) to maintain in the process testing lab. If a pH probe is used to monitor the titration then pH standards must also be kept on hand to calibrate the probe. In addition to the chemical requirements, precision volume measurement equipment for the sample and titration acid must be maintained in good working order and periodically calibrated.

Bench top (laboratory) autotitrators have been implemented to automate the actual titration test. However, most of the same issues associated with the standard "ABC" test are also present with the laboratory autotitrator. At a minimum, titration acid and pH standards as well as precision volume measuring equipment are still required. Most autotitrators are based on the SCAN (Scandinavian Pulp, Paper and Board Testing Committee) method. This method differs from the TAPPI standard "ABC" titration test in that formaldehyde and barium chloride are not used. Instead, a pH curve is generated

as a function of the added titration acid The inflection points on the titration curve are used to estimate the EA, AA and TTA values of the liquor sample. Problems can arise from difficulties in pinpointing the inflection points, especially for the AA. The inflection point locations can vary with changes in dead load concentrations, leading to erroneous concentration estimates. This effect is especially pronounced on the AA inflection point.

1.3 Specifications

The general specifications of the ***DURALYZER-NIR™*** laboratory liquor analyzer are contained in Table 1-2. Certain specifications may differ from Table 1-2 if unique installation and operating conditions require deviation from the standard setup.

Table 1-2. General Specifications.

Accuracy	EA: ± 0.50 lb/100gal as Na_2O
	AA: ± 0.75 lb/100gal as Na_2O
	TTA: ± 0.75 lb/100gal as Na_2O
	%TDS: ± 0.50
	%TDD: ± 0.50
	Lignin: ± 1.00 lb/100gal
	Na_2SO_4: ± 0.5 lb/100gal as Na_2O
Repeatabilty	± 0.2 % of full scale
Analyzer Internal Operating Temperature	40 °C
Sample Cuvette	Fused Silica, 1.0 mm path length
Tubing	Silicone
Analyzer Housing	NEMA 4X, 304 SS
Connections	RJ11 (remote support modem)
	Cat-5 RJ45 Ethernet (MODBUS TCP)
	Printer (RS232 9 pin D-sub)
	LCD touch screen panel (4 pin)
	RS422/485 MODBUS RTU (5 pin)
Power Input	120 - 240 VAC, 1.6A, 50/60 Hz

2 Principle of Operation

2.1 Concept

The ***DURALYZER-NIR™*** laboratory liquor analyzer is based on the implementation of NIR spectroscopy to determine the chemical composition of process liquors. This spectroscopic technique is a subset of a larger class of analytical techniques that fall under the category of optical spectroscopy. Figure 2-1 shows the electromagnetic spectrum, with the portion relating to optical spectroscopy expanded (NIR region highlighted with black arrow). Optical spectroscopic techniques have been used quite successfully for decades in a laboratory setting to analyze liquids, solids, and gases composed of a multitude of chemical species. In the past twenty five to thirty years, the development of miniaturized and durable electronic and optical components has allowed many of these techniques to be implemented in the process environment. Advancements in computational techniques and microcomputers have fueled the increased utilization of optical spectroscopy for qualitative and quantitative analysis in the process environment.

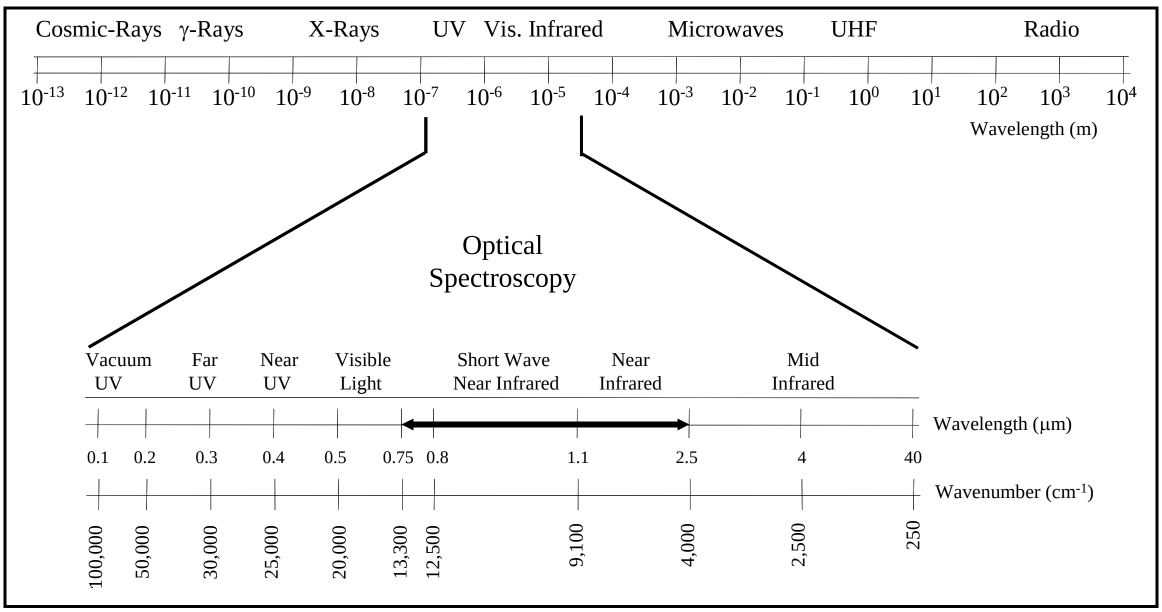


Figure 2-1. Electromagnetic spectrum related to optical spectroscopy.

NIR spectroscopy has many attractive features that make it ideally suited for process analysis. Key features include: minimal sample preparation, remote sensing through the use of fiber optic cables, and simple implementation using relatively inexpensive and highly robust components. There are a variety of optical attachments available to interface a NIR spectrometer to a sample for collection of spectral information. The most common attachments used for liquid analysis are a transmission cell and a sample cuvette. The basic configuration of a transmission cell is illustrated in Figure 2-2. Figure 2-3 depicts a sample cuvette.

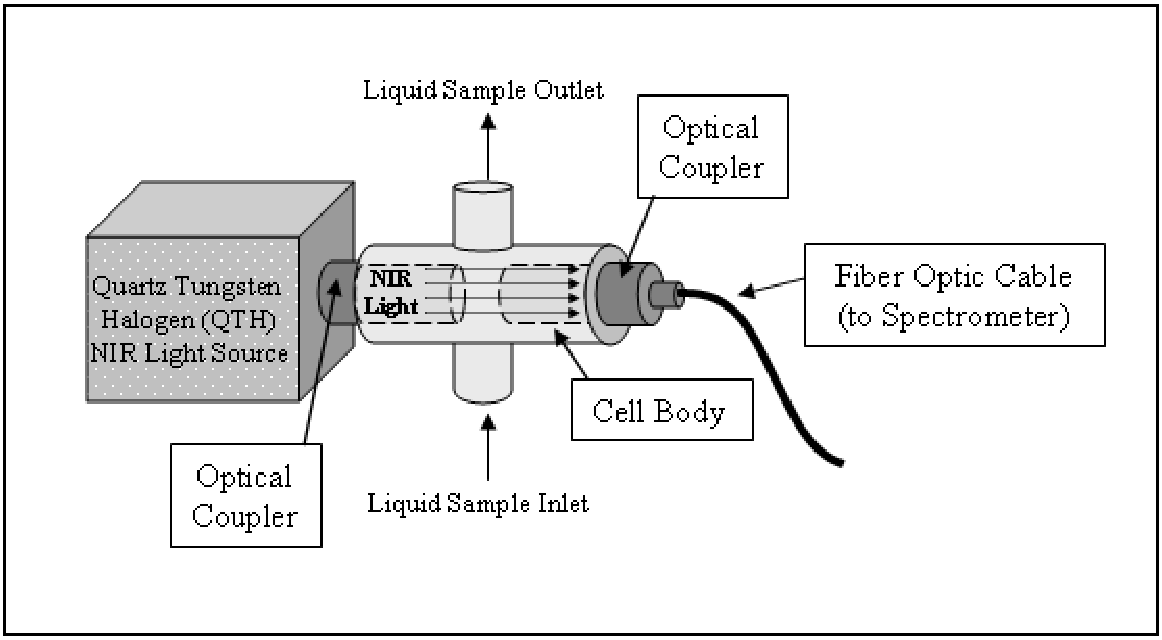


Figure 2-2. Basic transmission cell configuration.

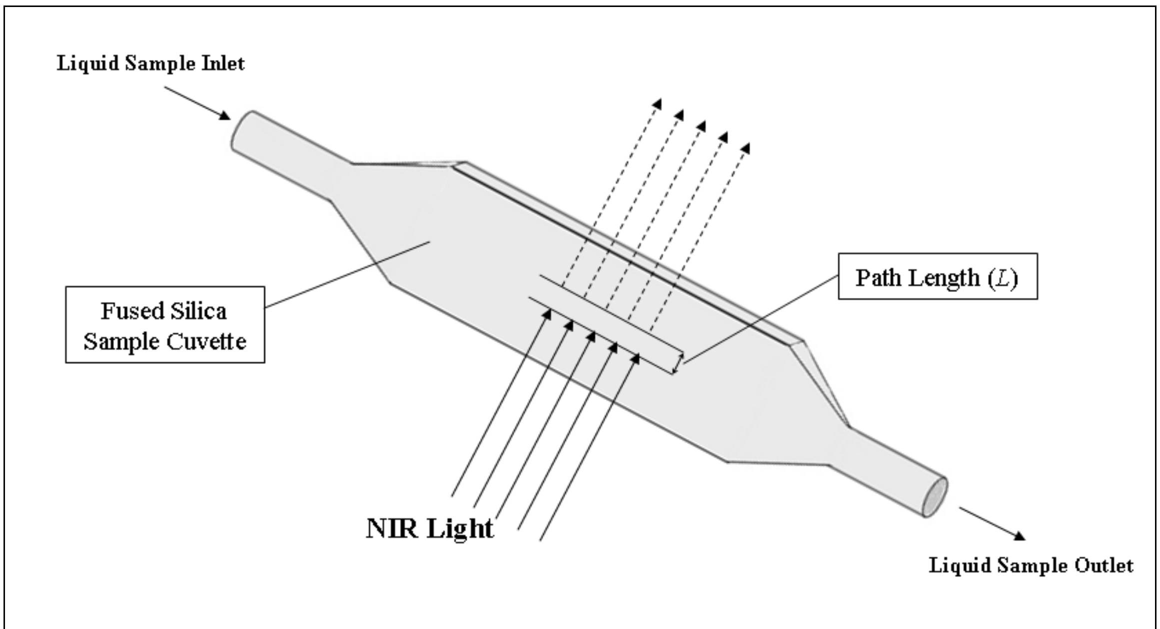


Figure 2-3. Sample cuvette.

The transmission cell allows NIR radiation to interact with the sample while isolating the light source, fiber optic cable, and spectrometer from the process. A typical transmission cell is composed of a body (with appropriate sample inlet and outlet connections) and a pair of optical couplers to deliver light to the sample and collect light after interaction with the sample. The optical couplers house a set of lenses to focus the radiation onto the tip of the fiber optic cable. The ends of the couplers in contact with the process sample have windows (usually sapphire) which provide a transparent optical path for the entering and exiting light as well as providing isolation from the process sample. Sapphire is

usually the material of choice for the coupler windows due to its combination of hardness, chemical and heat resistance, and inherent transparency over a broad range of wavelengths.

A sample cuvette functions as a sample cell and is used in a laboratory environment. Obviously, it would not hold up to the rigors of a process environment. Implementation of a cuvette requires a cuvette holder (shown in Figure 2-4) that allows the delivery of NIR light and collection of the transmitted light.

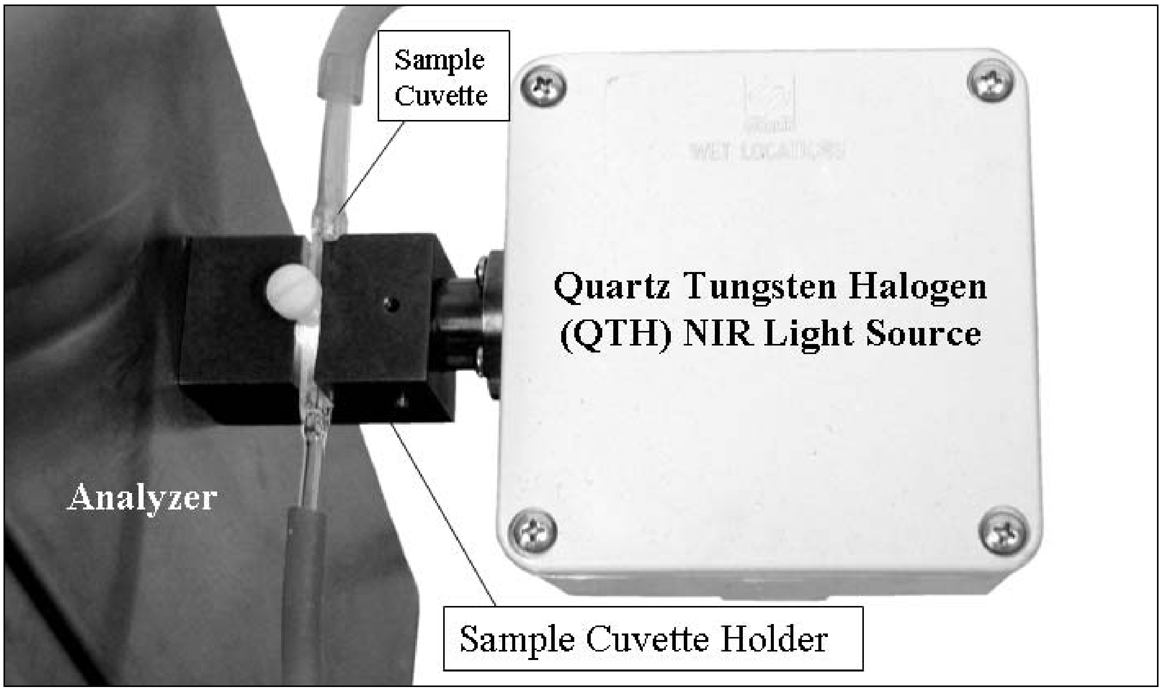
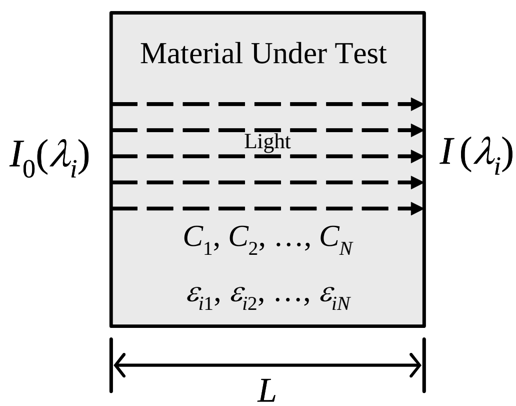


Figure 2-4. Sample cuvette holder.

Light interaction with the sample is described by the Beer-Lambert law (also referred to as Beer's law and Lambert-Beer's decay). This law states that absorbance is directly proportional to the concentration of the absorbing species. Details of this relation are shown in Figure 2-5. According to Beer's law, light intensity decays exponentially as it passes through the material. The rate of decay depends on the concentrations of the constituent species of the material and their corresponding absorption coefficients. The total amount of decay depends on the length of material the light crosses. Equations describing this phenomenon are displayed within Figure 2-5. Total absorption at a particular wavelength can be computed by applying a logarithm to the initial equation. The key observation to be made from this equation is that absorption (A) at a particular wavelength varies linearly with the concentrations of the constituent species (C_k). With the path length (L) fixed by the transmission cell, the absorption coefficients (ε_{ik}) depend only on the wavelength i and the molecular structure of species k in the material under test. Thus, the absorption will change only when the concentrations of the constituent species change. Additionally, absorption at a particular wavelength depends on the concentration of **all** of the species that make up the sample under test. This is a major drawback for single wavelength instruments that are used to analyze multi-component

materials. With these instruments, the best that can be done is to select an observation wavelength that is highly absorbed by the component of interest while simultaneously minimizing the absorption of the other components. Many times such a wavelength does not exist. This is an analogous situation to the application of conductivity for measuring effective alkali (EA) levels in white liquors. The white liquor conductivity is affected the most by the EA concentration but the sulfide and carbonate levels also have an affect on the conductivity. As a result, periodic recalibration of the conductivity meter is required to compensate for the effects of changing sulfide and carbonate levels. The ***DURALYZER-NIR™*** analyzers (laboratory and online) do **not** have these drawbacks or shortcomings since the measurement is taken over a continual range of wavelengths.



$$I(\lambda_i) = I_0(\lambda_i)\exp\left\{-\left(\sum_k^N \varepsilon_{ik} C_k\right)L\right\}$$

$$\frac{I(\lambda_i)}{I_0(\lambda_i)} = \exp\left\{-\left(\sum_k^N \varepsilon_{ik} C_k\right)L\right\}$$

$$A(\lambda_i) = -\ln\left\{\frac{I(\lambda_i)}{I_0(\lambda_i)}\right\} = \left(\sum_k^N \varepsilon_{ik} C_k\right)L$$

$I_0(\lambda_i)$ = Entering light intensity for wavelength λ_i

$I(\lambda_i)$ = Exiting light intensity for wavelength λ_i

$C_1, C_2, \ldots, C_N$ = Concentration of species 1, 2, …, N

$\varepsilon_{i1}, \varepsilon_{i2}, \ldots, \varepsilon_{iN}$ = Absorption coefficients for species 1, 2, …, N for wavelength λ_i

L = Path length of sample

$A(\lambda_i)$ = Total absorption for wavelength λ_i

Figure 2-5. The Beer-Lambert law.

The general concept is to utilize NIR spectroscopy (via a transmission cell or sample cuvette) in conjunction with software to determine the chemical composition (EA, AA, TTA, %TDS, and %TDD) of causticizing process liquor streams. Liquor samples are collected and prepared for analysis in the sample multiplexing & conditioning system. Analysis occurs in the transmission sample cell in which the liquor interacts with NIR light. The transmitted light is then collected via fiber optic cable and sent to the spectrometer. Chemical compositions are computed by passing the transmitted NIR light spectrum through the calibration model. The calibration model is based on sample spectra of known composition and constructed using a series of signal processing steps

and employing a variation of partial least squares (PLS) or other regression techniques. A simplified flow diagram of the regression analysis is contained in Figure 2-6.

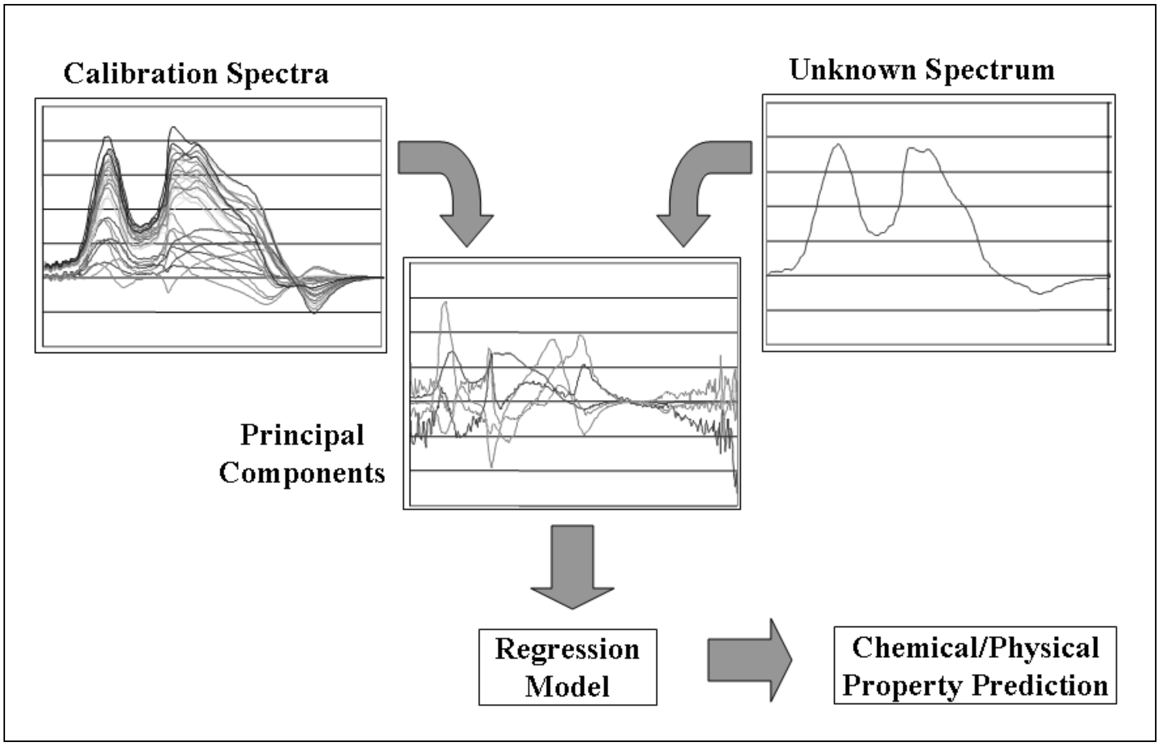


Figure 2-6. Simplified view of regression modeling and prediction.

2.2 Implementation

Direct implementation of NIR spectroscopy for laboratory liquor analysis requires an analyzer able to withstand long term use in the harsh lab environments of the pulping and recovery areas. The ***DURALYZER-NIR™*** laboratory liquor analyzer is housed in a 304 SS NEMA 4X enclosure which provides protection from corrosive agents, moisture, and dust. Attaching the sample cuvette holder and light source directly to the enclosure serves to eliminate the need for fiber optic cables and minimize the overall footprint (which is important due to the lack of work space in most pulp mill laboratories). Test results are displayed on the LCD touch screen panel and can also be printed via the accompanying thermal printer. Results can also be sent directly to the DCS through the MODBUS connection.

Delivery of the sample to the cuvette is simple. Silicone tubing is attached to the cuvette, with one end placed in the sample container and the other end attached to a syringe (or similar device). Currently, the analyzer is shipped with a 60 mL pipetting bulb. The design of the ***DURALYZER-NIR™*** laboratory liquor analyzer allows for custom arrangements. Figure 2-7 shows the syringe setup.

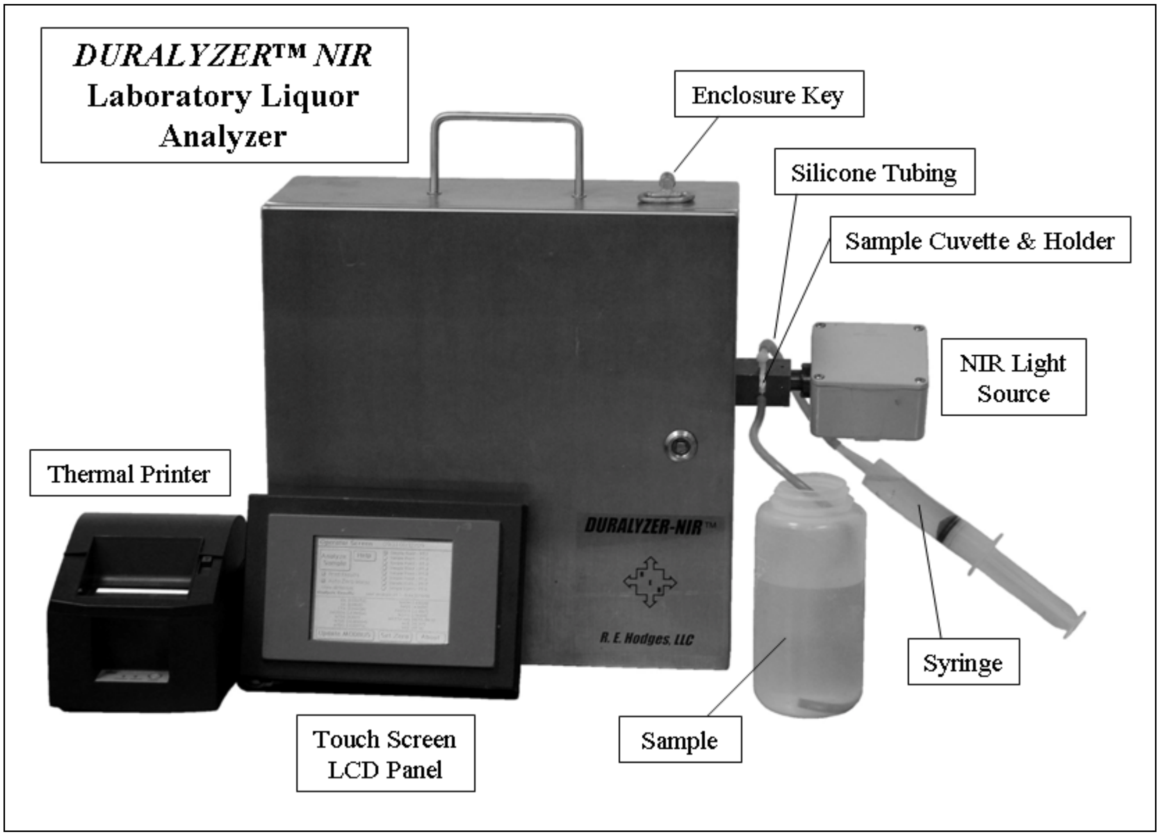


Figure 2-7. ***DURALYZER-NIR™*** laboratory liquor analyzer (syringe setup).

3 Installation

3.1 General

The ***DURALYZER-NIR™*** laboratory liquor analyzer is designed for straightforward installation (setup) while minimizing the associated costs (monetary and time). Highlights and key components are identified in Figure 2-7. The setup location should be in an area safely away from continual exposure to acid vapor. Care should be taken to avoid moisture exposure to the LCD panel and the thermal printer (panel and printer are attached via cables so this should not be too difficult). A detailed listing of environments to avoid is given in Appendix A.4 *Setup Cautions*. The analyzer comes delivered in a foam shell within a reinforced box that should be stored and used to ship the analyzer back to REH, LLC if the need arises for any major repairs.

3.2 Tubing Connections

Silicone tubing and a 60 mL rubber pipetting bulb are supplied to allow for drawing the sample into the cuvette and ejecting it after analysis (8-12″ for cuvette-to-bulb section and 18-22″ for sample bottle-to-cuvette section). This was previously mentioned and

shown in Figure 2-7. Alternatively, custom configurations can be employed. An example would be a "flow-through" arrangement, whereby the sample is poured into a funnel, flows downward through the cuvette to a collection container. As the sample is flowing, the tubing section downstream of the cuvette would be temporarily clamped for the duration of the analysis.

3.3 Air and Water Requirements

Instrument air is **not** required for operation. Mill water is required for flushing out the sample cuvette and occasional spectrometer baseline referencing. Deionized water is **not** needed unless the mill water is of unusually poor quality. If mill water quality is an issue, a suitable solution will be proposed.

3.4 Power and Wiring Connections

The analyzer is equipped with a detachable power cord connectable to any standard (120V, 60 Hz, single phase 10A) outlet with line, neutral, and ground connections. The thermal printer is connected to the analyzer via the RS232 port and is equipped with its own power unit and power cord which also connects to any standard 3 prong outlet. Further details concerning the thermal printer are located in Appendix A.9 *Printer Details*. The LCD touch screen cable is plugged in to the 4-pin connector located next to the RS232 printer connection. A MODBUS RTU 5-pin connection port is also available externally. All external connection locations are on the side of the analyzer opposite the cuvette and exhibited in Figure 3-1.

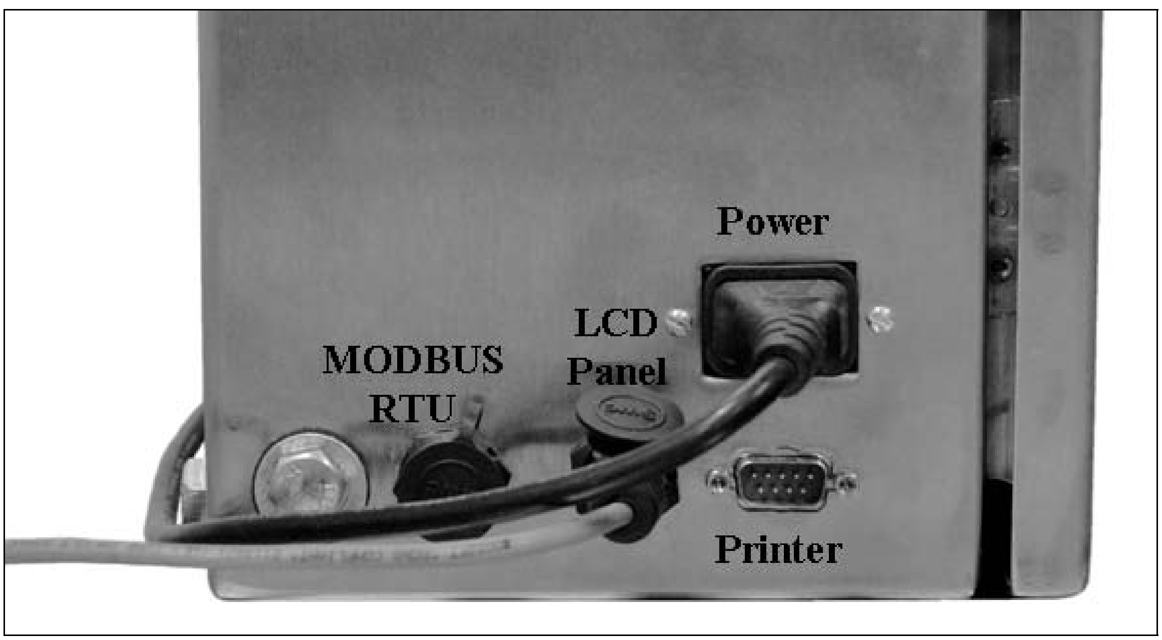


Figure 3-1. External connection locations.

Two internal connections are also present. Remote modem support is provided through the RJ11 connection, enabling software updates and an ability to assist mill personnel in

troubleshooting (if the need arises). A Cat-5 RJ45 ethernet connection is available for MODBUS TCP. The internal connections as well as key internal components are identified in Figure 3-2.

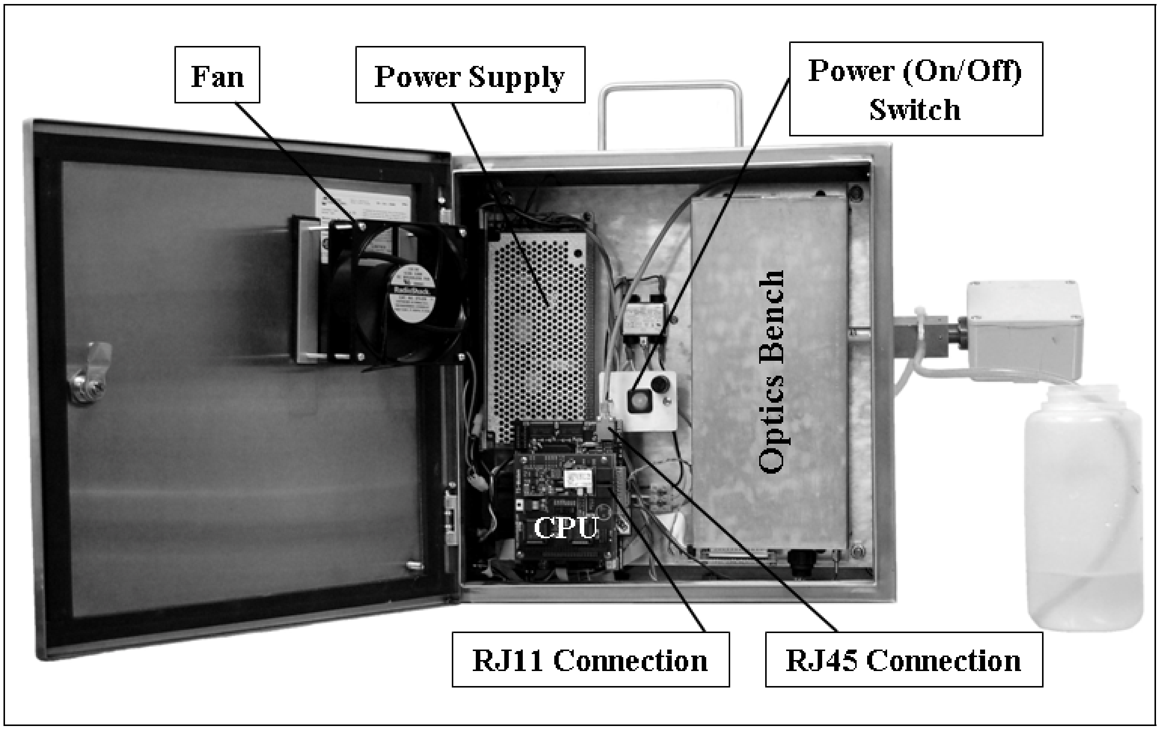


Figure 3-2. Internal connections and components.

4 Operation and Maintenance

4.1 General

The ***DURALYZER-NIR™*** laboratory liquor analyzer has been designed for ease of operation and minimization of short and long-term maintenance requirements. This has a net effect of minimizing the overall cost of ownership. Scheduled operation and maintenance requirements have been kept to a minimum. These include annual bulb replacement in the light source enclosure and occasional acid cleaning of the sample cuvette. Periodic validation with lab tests should be performed as well. Unscheduled maintenance has been greatly reduced by minimizing the system component count. As with any sophisticated hardware, common sense care will aid in extending the lifetime of the analyzer. Follow standard laboratory safety protocols when using the analyzer, such as wearing protective eyewear and clothing.

4.2 Startup

Startup is straightforward, entailing setup tasks and power up. In some cases, R.E. Hodges, LLC personnel can be on hand to perform and oversee startup operations. The startup procedure is outlined in Table 4-1. Figure 4-1 shows the LCD screen when power is activated.

Table 4-1. Startup Procedure

1.	Identify a safe, convenient, and hazzard free location (take into account "Setup Cautions" listed in the Appendix)
2.	Unpack and arrange accessories (printer, etc.)
3.	Connect all power cables and communication cords
4.	Attach tubing to cuvette and pipetting bulb
5.	Secure cuvette in holder with set screw
6.	Turn on analyzer and close SS enclosure with key
7.	Allow analyzer to be powered on for a MINIMUM of 2 hours before proceding with any analysis activities (internal components need to reach thermal equilibrium and stabilize)

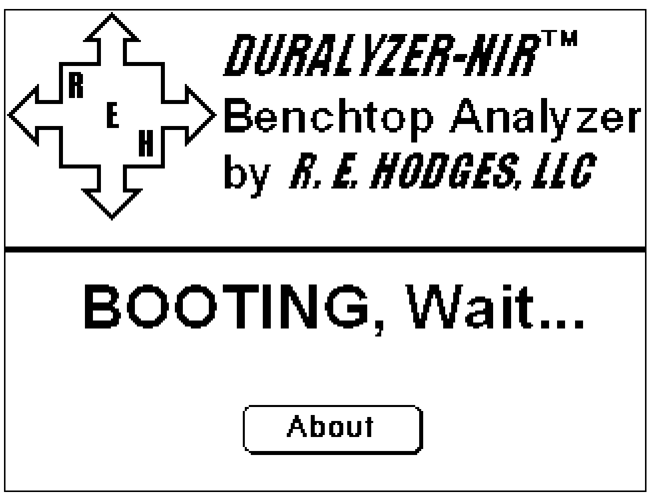


Figure 4-1. LCD “BOOT” screen.

4.3 Normal Operation

Normal operation of the ***DURALYZER-NIR™*** laboratory liquor analyzer is comprised of two steps: (1) deliver sample to cuvette and (2) analyze the sample. A step by step normal operating procedure is in Appendix A.5 *Normal Operating Procedure*. Before performing any analysis, the analyzer **must** be powered on for a **minimum of 2 hours** (12 or more hours would be ideal). Once powered “on”, the analyzer should only be

turned off when it is being moved to another location or serviced. Initially, operation will be outlined using the "default" settings from REH, LLC. Procedures concerning calibration model tuning and configuration settings are detailed in subsequent sections. The LCD should reflect Figure 4-2 at this point, showing the *Operator Screen*.

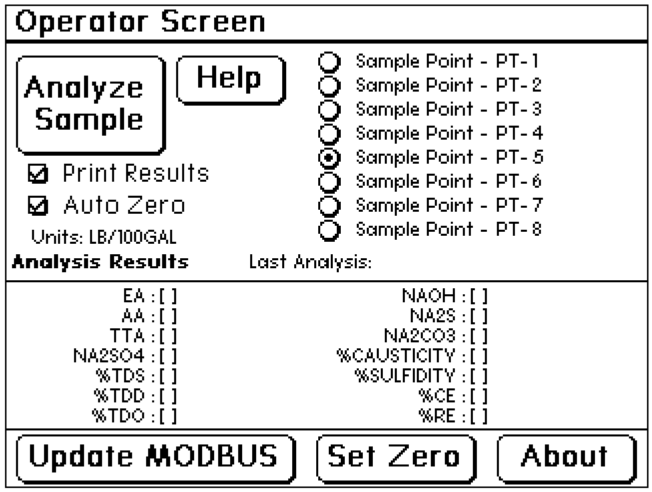


Figure 4-2. *Operator Screen.*

Before initiating sample analysis, a water "reference" or "baseline" is taken. Fill a sample container with water (typically 200 mL of water in a 250 mL Nalgene® bottle). Fully compress (squeeze) the pipetting bulb and then insert the silicon tubing (pickup tube) into the water. Draw the water into the cuvette by slowly releasing the bulb and then let it hang freely. Select "Set Zero" on the LCD touch screen (finger or stylus may be used). In addition to setting a baseline, setting the water zero also ensures the wavelength axis of spectrometer (specifically the monochromator in the optics bench) is properly aligned. The corresponding screens for setting the zero are depicted in Figure 4-3. Scanning is delayed 120 seconds to ensure the water is stabilized (the delay can be bypassed by selecting "continue"). The message at the bottom of the screen will change when the water is being scanned (spectrum acquired).

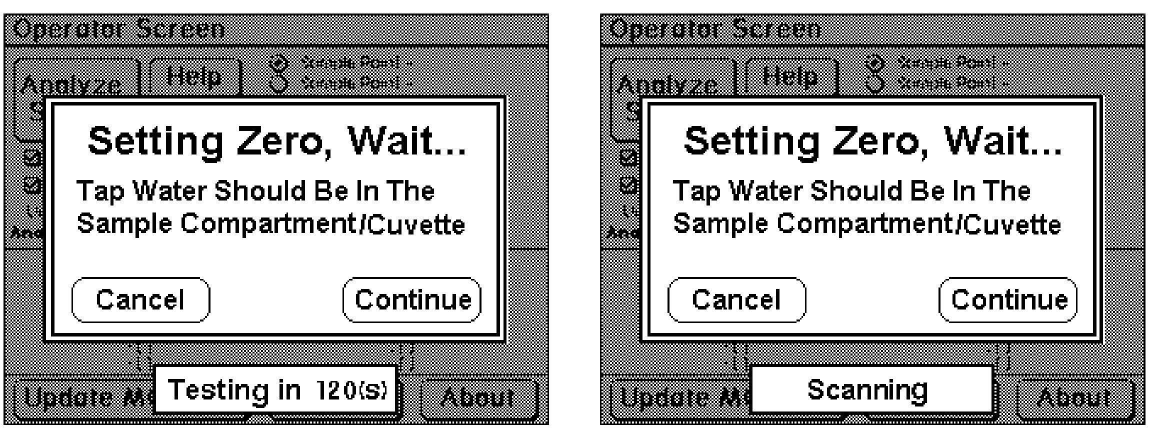


Figure 4-3. "Set Zero" screens.

The "zero" should be set once per day, plus every time the analyzer is turned on (once stabilized), and after acid cleaning the cuvette. This can be done manually (outlined above) or by selecting the "auto zero" check box (see Figure 4-2). When sitting idle, water is to be in the cuvette at all times. The "auto zero" function takes advantage of this, setting the zero every 2 hours.

Before analyzing a liquor sample, place the silicon tube in a sample waste bottle and eject the water. Hold the pipetting bulb in an upright (elevated) position and squeeze. Do this several times to thoroughly evacuate the water (note: the silicon tubing should be held while doing this to avoid the possibility of uncontrolled spray). Fully compress the pipetting bulb and place the silicon tubing in the liquor sample. Draw the liquor into the cuvette by slowly releasing the bulb and then let it hang freely. Select the sample point for testing and "Analyze Sample" on the Operator Screen (Figure 4-2). The current status of the LCD screen is reflected in Figure 4-4. Scanning is delayed 120 seconds (default value) to ensure the sample is stabilized (the delay can be bypassed by selecting "continue"). The message at the bottom of the screen will change when the sample is being scanned (spectrum acquired).

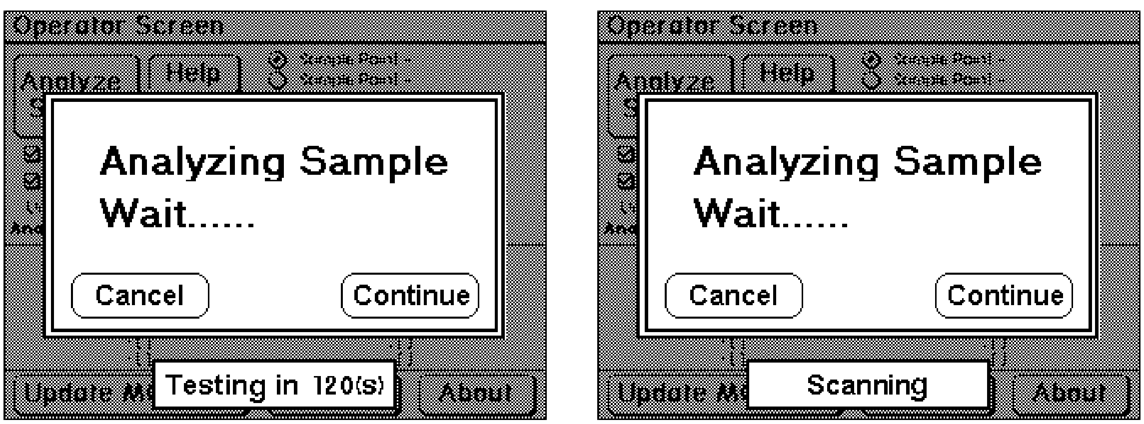


Figure 4-4. "Analyze Sample" screens.

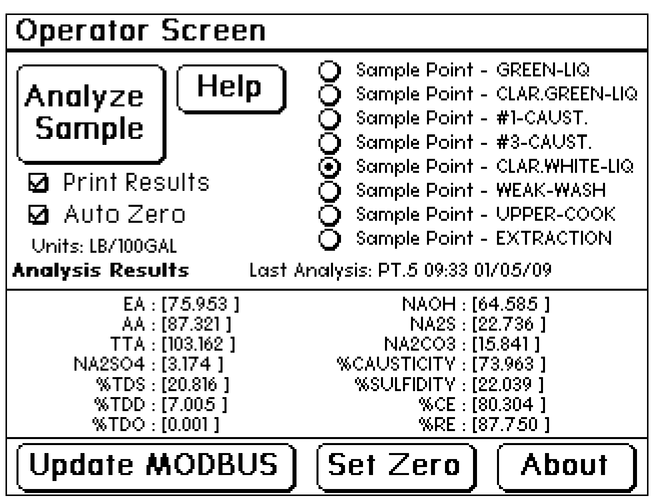


Figure 4-5. Analysis results.

After scanning is complete, the Operator Screen will update to display the analysis results. Figure 4-5 is an example for a clarified white liquor sample. Checking the "Print Results" box enables the results to be printed out via the thermal printer. If the MODBUS communications interface is being employed, select the "Update MODBUS" option on the screen to send the results to the appropriate registers. Flush the sample cuvette with water and eject into the waste bottle. Draw water into the cuvette, letting it remain until the next round of liquor testing. Items such as labels and display units can be customized (edited) to suit the user. Customization options are detailed in the following section.

Periodically, the sample cuvette needs to be cleaned with 1.0 N or 0.1 N HCl acid. The frequency of cleaning is initially suggested to be once per day. However, it may end up being more infrequent depending on the scaling conditions and the number of samples being analyzed each day. Acid is drawn into the cuvette in the same manner as water or a liquor sample and allowed to sit in the cuvette for 2 minutes. The acid is then ejected into the waste collection bottle and flushed with water. Make certain the waste bottle has been thoroughly washed with water before ejecting the acid to avoid H_2S generation.

As a general guideline, each sample point should consistently be tested in the same 10 °C window for optimal results (i.e., maintain consistent conditions under which each sample is tested). If this is a recurring problem, samples can be capped and placed in a water bath. For example, place the capped samples in a large container in a sink, fill with tap water, and allow the water to overflow for 2 minutes. Remove the samples from the water bath and proceed with testing. This is detailed in the *Normal Operating Procedure* in Appendix A.5.

4.4 Settings

4.4.1 Accessing the *Main Menu*

Additional information and the ability to customize the analyzer settings can be accessed by selecting "About" on the *Operator Screen* (Figures 4-2, 4-5). At this point, an informational screen appears (Figure 4-6) displaying the company logo and the telephone number for support. Selecting "OK" will return the user to the *Operator Screen*. Touching the trademark (gray shaded region in Figure 4-6) will access a virtual keyboard on the LCD screen (Figure 4-7).

The "QWERTY" virtual keyboard allows upper case letters, numerals, and some standard symbols to be input. Spacebar and lower case functionality is not available. The dash "-" may be used in lieu of a "space". To gain access to the *Main Menu* (Figure 4-8) type in the password and select "Enter". The default password is "ADMIN" and can be changed by the end user.

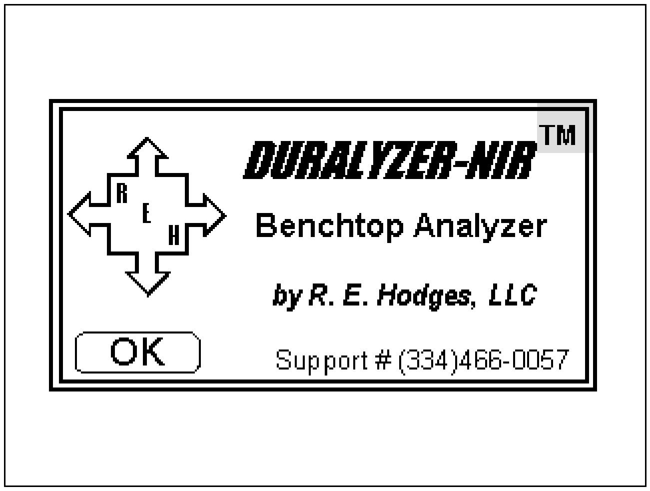


Figure 4-6. "About" screen.

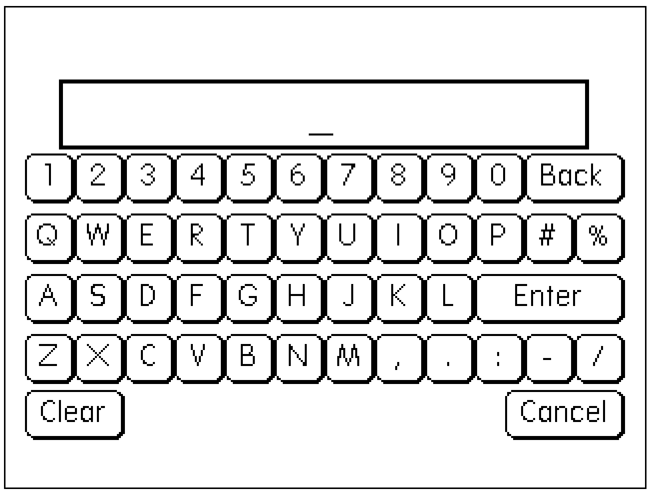


Figure 4-7. LCD touch screen keyboard.

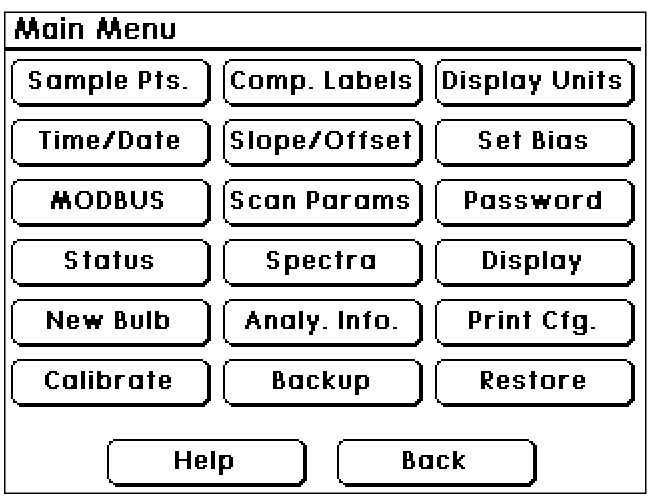


Figure 4-8. *Main Menu* screen.

The *Main Menu* is the access point to 18 submenus which serve to enhance functionality and provide customization. To return to the *Operator Screen*, simply touch "Back" and then "OK" when the "About" screen (Figure 4-5) appears. The submenus can be categorized under four headings: (1) labeling, (2) configuration, (3) informative, and (4) model tuning. Table 4-2 shows a summary of the submenus accessible from the *Main Menu* displaying the name, description, and category.

Table 4-2. *Main Menu* Summary.

Submenu	Description	Category
Sample Pts.	Define each sample point label	Labeling
Comp. Labels	Custom label each measured component	Labeling
Display Units	Define units for each sample point	Configuration
Time/Date	Change analyzer time and date	Configuration
Slope/Offset	Access component curve,slope, and offset	Model Tuning
Set Bias	Adjust biases to match lab tests	Model Tuning
MODBUS	Configure MODBUS/TCP parameters	Configuration
Scan Params	Change scan count and time delay	Configuration
Password	Change current password	Configuration
Status	Display internal analyzer sensor readings	Informative
Spectra	Display spectral signature of current sample	Informative
Display	Adjust brightness and contrast of LCD panel	Configuration
New Bulb	Check for saturation on a new bulb change	Informative
Analy. Info.	Display model#, serial#, and software version	Informative
Print Cfg.	Print analyzer configuration	Informative
Calibrate	Perform analyzer calibration function	Model Tuning
Backup	Backup current configuration	Configuration
Restore	Restore last saved configuration	Configuration

4.4.2 Labeling Submenus

Selecting "Sample Pts." enables customized labeling of the sample points. Each sample point can be labeled with a custom description (label) that is displayed on the *Operator Screen* and the analysis print out. The sample point number is displayed to the left of the editable label. Touch the button to edit the corresponding sample point label. Figures 4-9, 4-10, and 4-11 show the *Sample Point Labels* screen progressing from no sample points labeled to eight sample points labeled. The default labels are "PT-1", "PT-2", ···, "PT-8". For example, select the sample point 1 label button (box) and the virtual keyboard will appear (Figure 4-7), use "Clear" to remove the previous label, type in "GREEN-LIQ" (or the label of your choice) and select "Enter" (Figure 4-10). Continue labeling the remaining sample points in the same fashion (Figure 4-11) and touch the "Update" button when finished. To return to the *Main Menu*, select "Back".

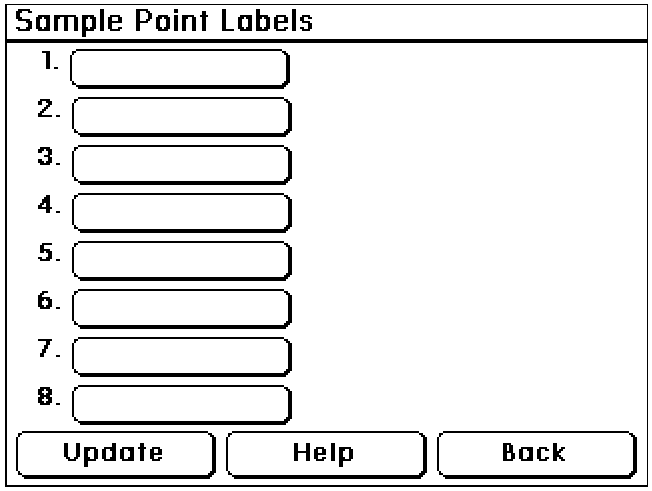


Figure 4-9. Blank *Sample Point Labels* screen.

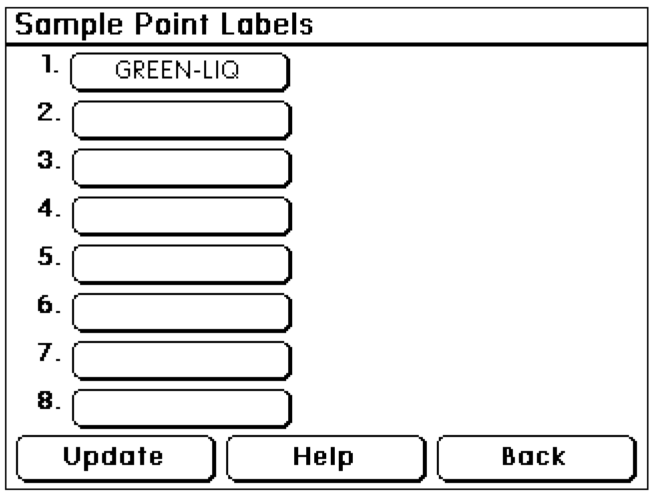


Figure 4-10. Sample Point 1 labeled as “GREEN-LIQ”.

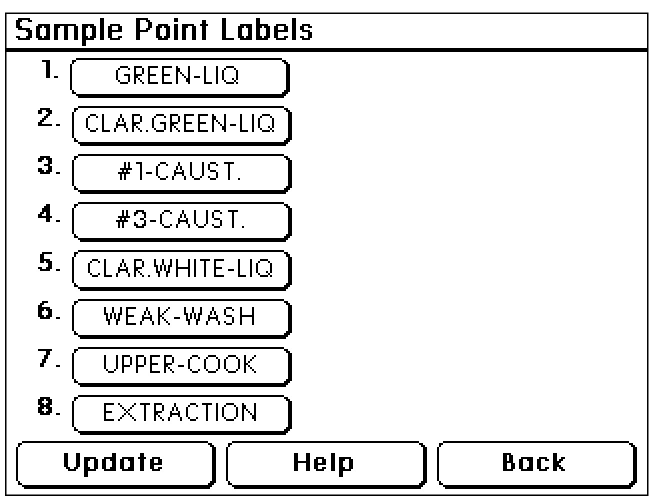


Figure 4-11. All sample points labeled.

From the *Main Menu*, changing the component labels is accomplished by selecting "Comp. Labels". Each measured component can be labeled with a custom description that is displayed on the *Operator Screen* and the analysis print out. The measured component label is displayed to the left of the button (box). Touch the button to edit the corresponding component label. The procedure is analogous to editing the sample point labels. Figure 4-12 shows the *Component Labels* screen with the default labels. As an example of customized component labels, the "EA", "AA", "TTA" can be represented as "A", "B", and "C" (in reference to the standard titration test) and is depicted in Figure 4-13.

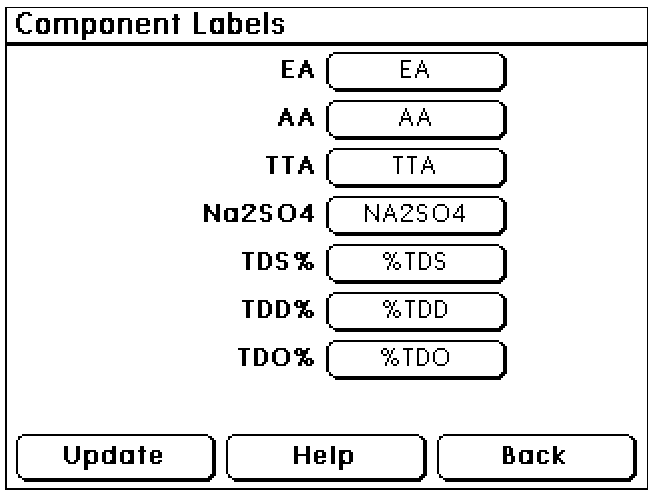


Figure 4-12. *Component Labels* screen with default values.

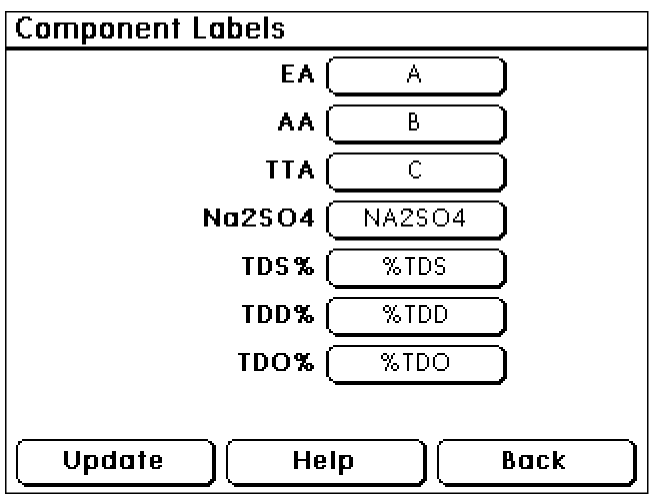


Figure 4-13. Customized EA, AA, and TTA labels.

4.4.3 Configuration Submenus

By default, measured component values (that are not reported on a percentage basis) are reported in lb/100gal as Na_2O. This can be changed by choosing "Display Units" on the *Main Menu*. Each measured component (value) can have custom units defined. Referring to Figure 4-14, the left column contains the unit labels for each measured

component. The right column contains the corresponding conversion factor to convert from lb/100gal as Na_2O to the desired units. The procedure is analogous to editing the sample point labels. Touch the appropriate editable box (button) and enter the new values. Some typical units and conversion factors are given in Table 4-3. An example of editing the measured units from lb/100gal as Na_2O to lb/ft^3 as Na_2O is shown in Figure 4-15.

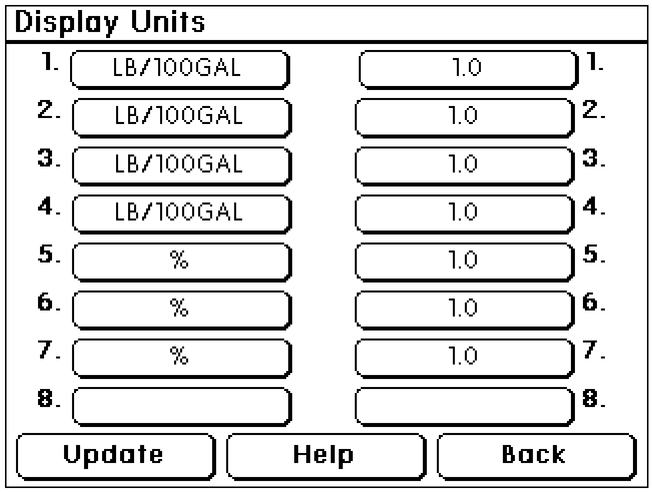


Figure 4-14. *Display Units* screen with default units (lb/100gal as Na_2O).

Table 4-3. Typical Units and Conversion Factors.

Display Units	Conversion Factor
lb/100gal as Na_2O	1.000
lb/10gal as Na_2O	0.100
lb/gal as Na_2O	0.010
lb/ft^3 as Na_2O	0.074805
g/L as Na_2O	1.198262
mL/HCl (10 mL sample)	0.38658
mL/HCl (5 mL sample)	0.77316

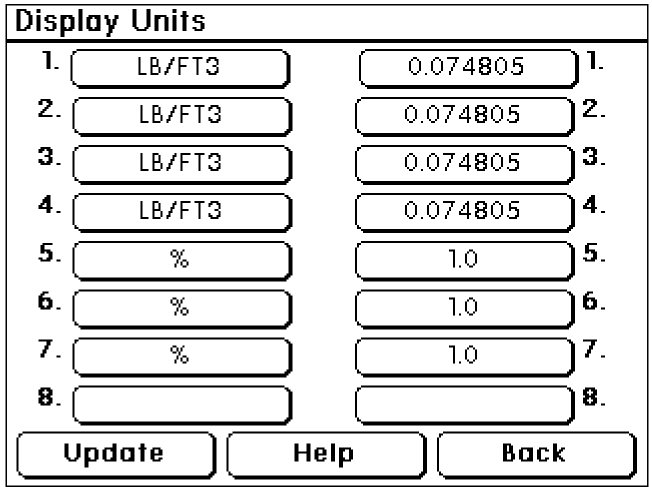


Figure 4-15. Display units in lb/ft^3 as Na_2O.

Setting or adjusting the analyzer time and date that is displayed on the *Operator Screen* and the analysis print out is performed by selecting "Time/Date". The *Time and Date Adjustments* screen would then appear (Figure 4-16); touch the appropriate box to edit. Time (military, hh:mm) and date (dd/mm/yy) must be entered in the exact format shown on the screen for the changes to take effect. For the changes to be accepted, both the time and the date must be entered. For example, setting the time and date to 12:01 January 5, 2009 is shown in Figure 4-17. Press the "Update" button for the changes to take effect and then the "Back" button to return to the *Main Menu*.

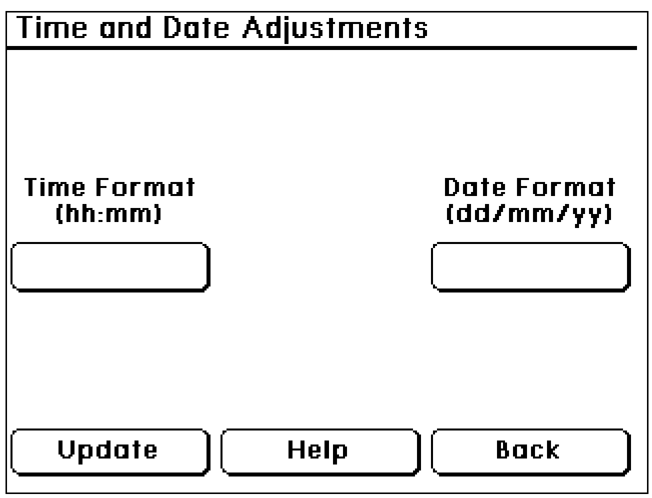


Figure 4-16. *Time and Date Adjustments* screen.

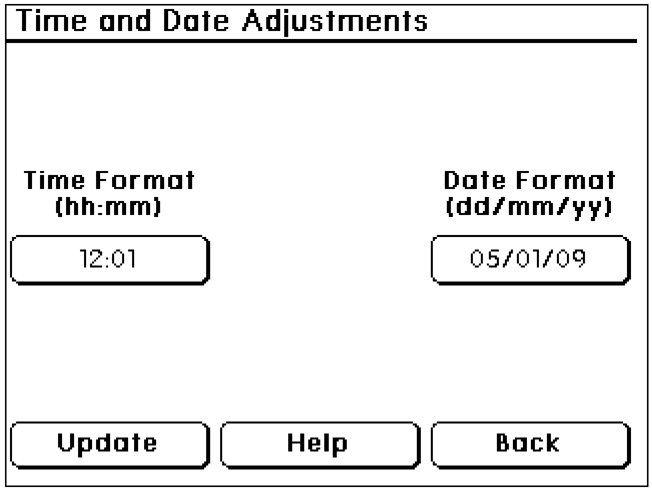


Figure 4-17. Example of setting the time and date.

If the MODBUS communications interface is to be utilized, configure it by selecting "MODBUS" on the *Main Menu*. This will pull up the *MODBUS Configuration* screen, shown in Figure 4-18 with example settings. The settings on this screen are used to configure the MODBUS TCP interface to the analyzer. Simply touch the box to be edited and enter the new value using the virtual LCD keyboard. Entries must conform to the format displayed above each box. A detailed description of this interface and how to configure it is documented in the Appendix A.11 *MODBUS*.

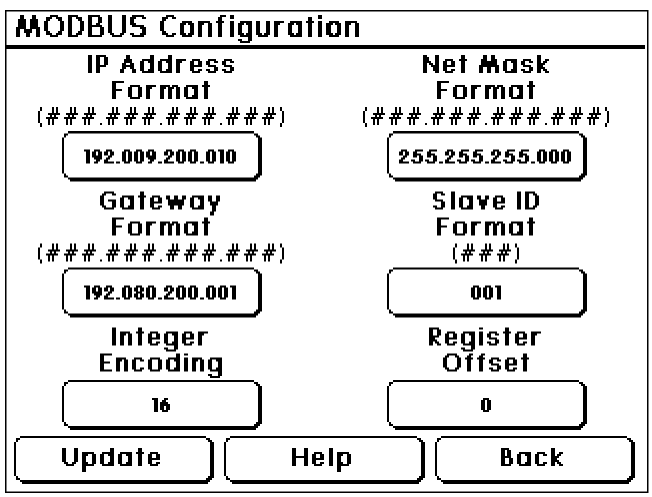


Figure 4-18. *MODBUS Configuration* screen.

Adjusting the number of spectral scans to be averaged and the settling time for sample analysis is accomplished by selecting "Scan Params" on the *Main Menu*. Custom scan parameters can be specified for each sample point. Figure 4-19 shows the *Scan Parameters* screen with the default values of "1" and "120" for each sample point. The left column specifies the number of scans (spectra) to average for a sample analysis and the right column specifies the settling time (in seconds) for the sample once it has been drawn into the cuvette. Typically, for white and green liquor, 2 to 3 scans are averaged and a settling time of 120 seconds is used. Black liquor samples may need 5 or more

scans to be averaged and 180 to 300 seconds of settling time. The settling behavior and nature of each liquor sample will dictate the optimal scan parameters used at a particular mill. Figure 4-20 illustrates custom scan parameters applied to all eight sample points.

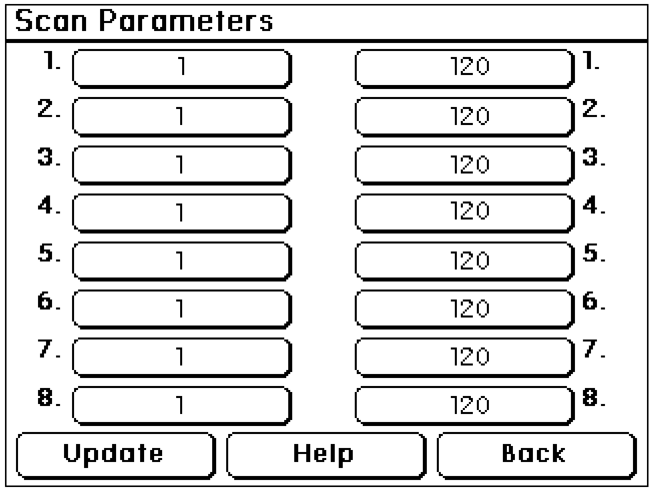


Figure 4-19. *Scan Parameters* screen with default values for each sample point.

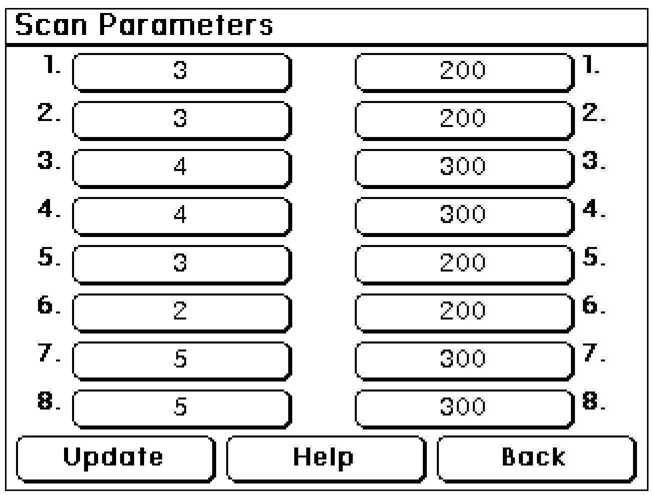


Figure 4-20. Custom scan parameters applied to each sample point.

Selecting the “Password” option form the *Main Menu* allows the user to change the current password. The analyzer ships with the default password “ADMIN”. Consider the case where the current password is “TIGERS” (Figure 4-21). The current password is shown on the left and the new password is entered on the right (by selecting the box and entering the new password on the virtual keyboard). If the new password chosen is “LIQTEST”, the *Change Password* screen will appear as shown in Figure 4-22. Press the “Update” to save the new password.

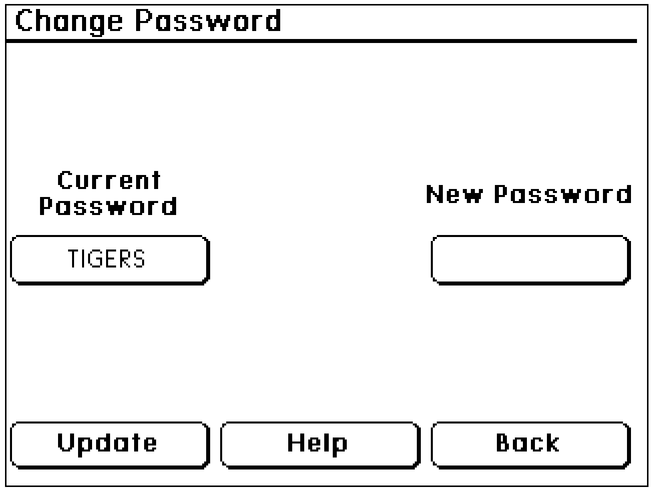


Figure 4-21. *Change Password* screen where the current password is “TIGERS”.

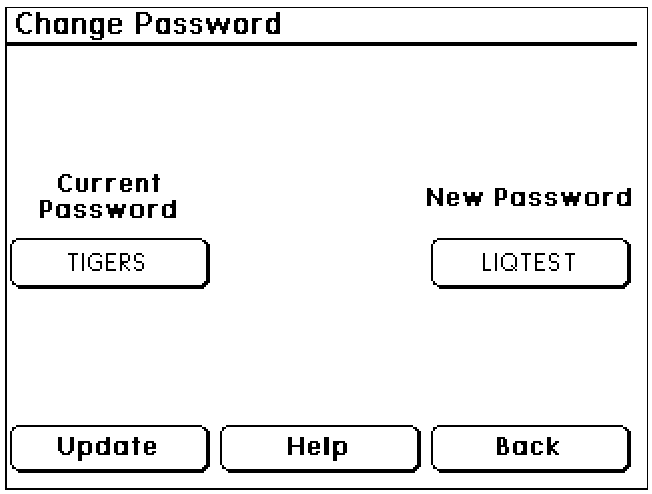


Figure 4-22. Password is changed to “LIQTEST”.

The “Display” option on the *Main Menu* allows the user to adjust the contrast and backlighting of the LCD screen. Figure 4-23 shows the *Contrast & Backlight Adjust* screen where the adjustments are made by manipulating the appropriate touch screen sliders. To accept the changes, press “Save”, otherwise select “Cancel”.

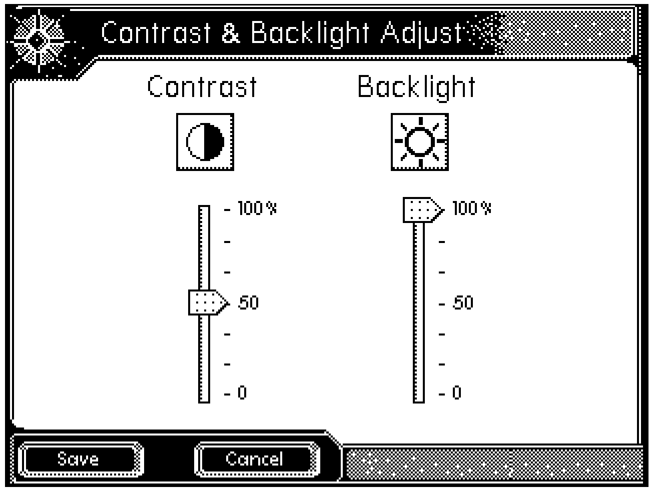


Figure 4-23. *Contrast & Backlight Adjust* screen.

In order to save a configuration, “Backup” is selected on the *Main Menu*. Selecting this option triggers the message in Figure 4-24. When making configuration changes, it is recommended to save (backup) after each change. If it becomes necessary to return to the last saved configuration, select the “Restore” option on the *Main Menu*. The screen (Figure 4-25) will give the option of canceling the operation or to continue and overwrite the current configuration.

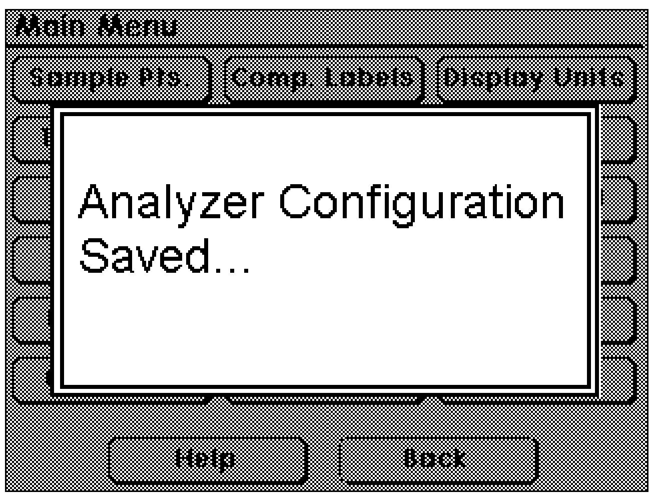


Figure 4-24. Saving the current analyzer configuration.

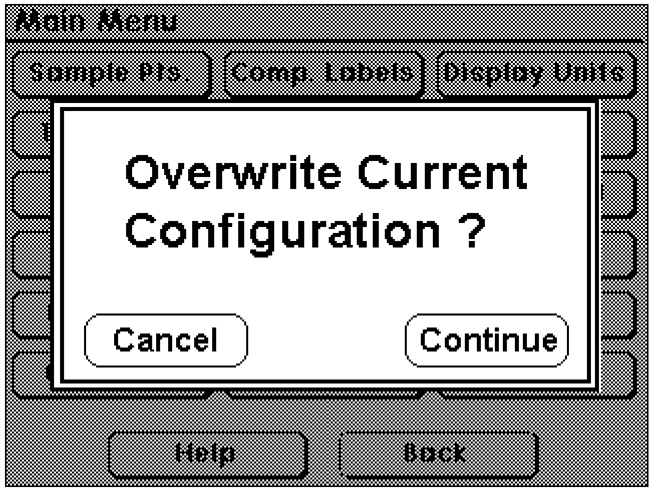


Figure 4-25. Utilizing the "Restore" function.

4.4.4 Informative Submenus

The analyzer internal sensor values are viewed through the "Status" option on the *Main Menu*. The screen (Figure 4-26) shows the current internal sensor values. The 5V source should read from 4.8 to 5.2 volts and the TEC set point should be between 1.48 and 1.51 volts. Detector bias should be less than 0.1 volts and the internal temperature should be below 35 °C. If the parameters are within the specified range, the analyzer is operating normally.

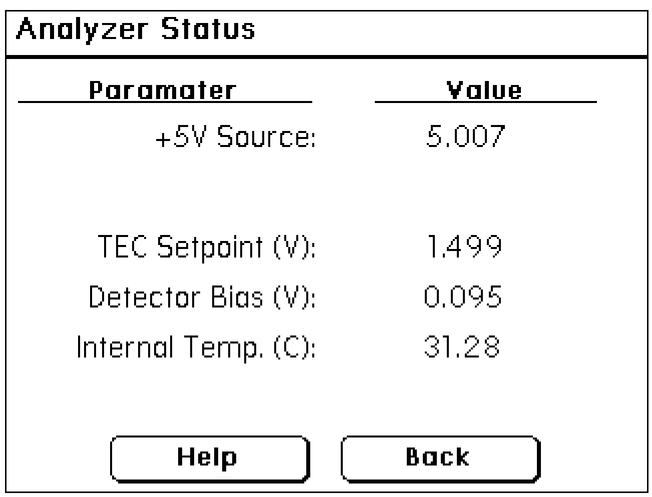


Figure 4-26. *Analyzer Status* screen.

A graphical display of the spectral signature of the last sample analyzed (scanned) is available by pressing "Spectra" on the *Main Menu* screen. The spectra can be displayed in terms of Transmission % or Absorbance % by selecting the appropriate option on the left side of the screen. A typical transmission water spectra is displayed in Figure 4-27.

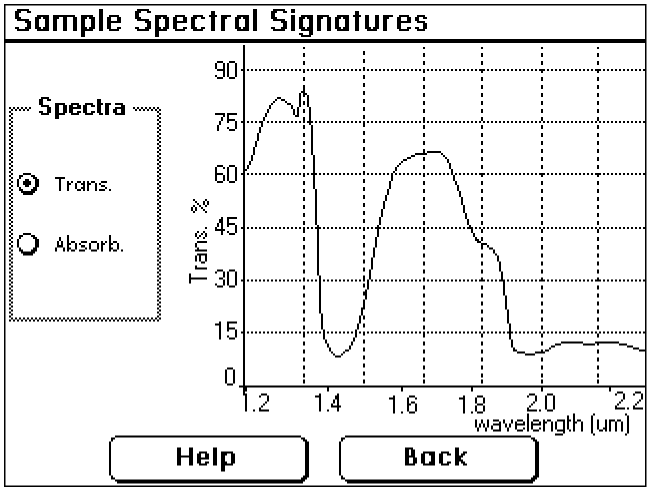


Figure 4-27. Typical transmission water spectrum.

Replacement of the NIR light source (QTH light bulb with reflective coating) may require a slight adjustment after installation. The "New Bulb" function on the *Main Menu* will access the *New Bulb Adjustment* screen. After the bulb has been replaced, draw water into the sample cuvette. Select the "Trigger Scan" option on the left side of the screen to check if the new bulb saturates the detector. Figures 4-28 and 4-29 show a water scan without saturation and a water scan in which the detector is saturated. An unsaturated water scan (Figure 4-28) indicates the replacement bulb is positioned and functioning correctly. If the water scan resembles the spectrum shown in Figure 4-29 (top portion of the two peaks are "cut off" meaning transmission has reached 100%), the detector is saturated. Detector saturation occurs even if only one wavelength is 100%. The desired transmission % is between 75 and 85 for the highest spectral peak. The remedy for detector saturation is to slightly reposition the bulb (by rotation and/or relative location in the light source holder). Details on removal and replacement of the light source are discussed in Section 4.5 *Maintenance* and Appendix A.10 *NIR Light Source Replacement*.

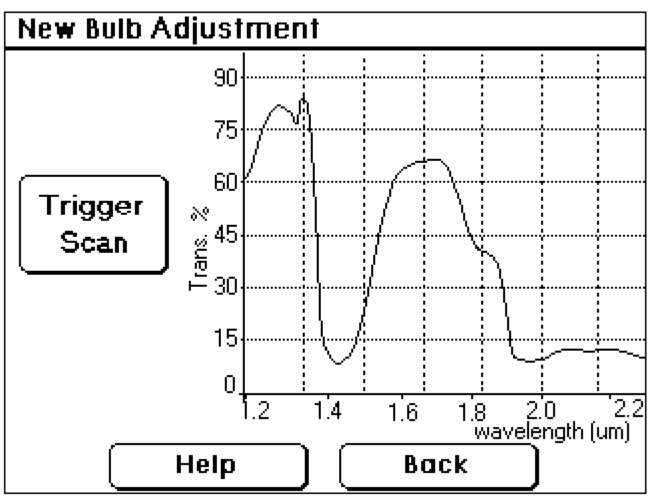


Figure 4-28. Unsaturated water spectrum.

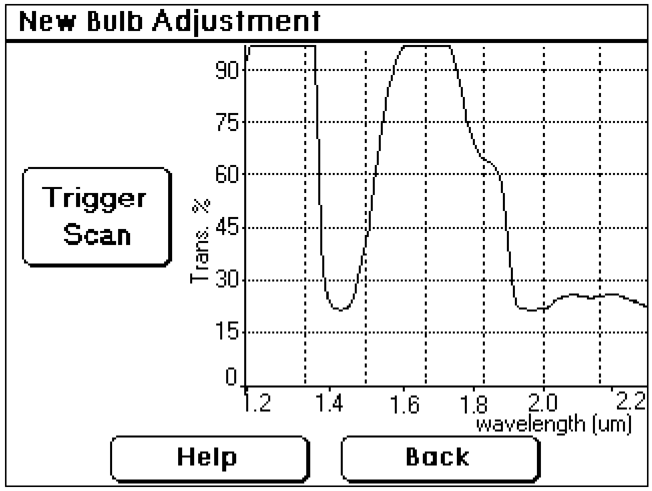


Figure 4-29. Saturated water spectrum.

Basic analyzer information such as the model number, serial number, and software version is displayed by selecting "Analy. Info." on the *Main Menu* screen. A sample display is shown in Figure 4-30. The "Print Cfg." option prints out a hard copy summary of the analyzer configuration. Selecting this option will briefly cause a notification screen (Figure 4-31) to appear.

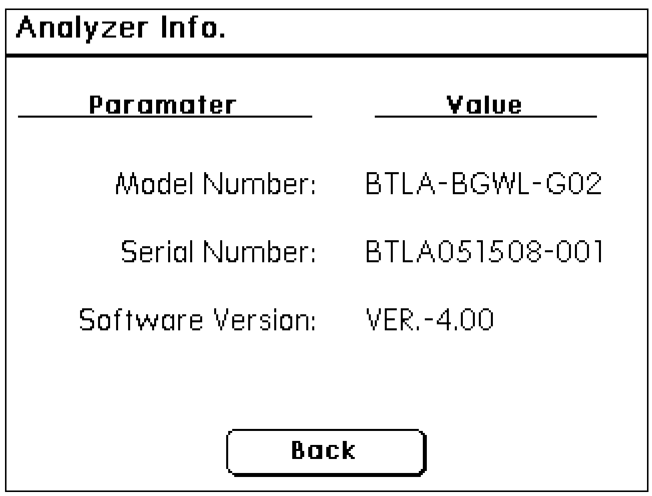


Figure 4-30. *Analyzer Info.* screen.

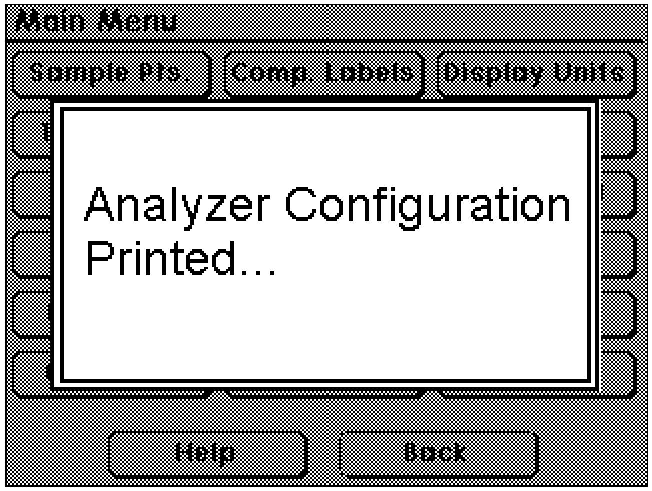


Figure 4-31. "Print Cfg." notification screen.

4.4.5 Model Tuning Submenus

The ***DURALYZER-NIR™*** laboratory liquor analyzer has seven standard calibration (predictive) models for analyzing green, white, and black liquor samples. The calibration models have been developed through a combination of signal processing steps and mathematical regression techniques. In some cases, the models need to be tuned by utilizing a calibration curve. The seven models are as follows: EA (REA for black liquor), AA (RAA for black liquor), TTA, Na_2SO_4, %TDS, %TDD, and %TDO. Each model will have a separate calibration curve and be represented by a second order polynomial. The curve "tunes" (adjusts) the model in a least squares manner to agree more precisely with lab tests (measurements). There are two methods for determining calibration curves: manual and automated.

Calibration curve parameters can be computed (through the use of a spreadsheet) and entered manually on the *Component Curves, Slopes and Offsets* screen (accessed by selecting "Slope/Offset" on the *Main Menu*). The default values for the calibration curves are shown in Figure 4-32. Each particular component calibration model has a box for entering the "Curve", "Slope", and "Offset" parameters. Due to the water zero function (sets the baseline), the offset will always be zero. Alternatively, the "Calibration" function on the *Main Menu* can be used to bypass manual computation and parameter entry. This is accomplished on the *Calibrate Curve, Slope and Offset* screen (Figure 4-33). Values for determining calibration curves for EA, AA, TTA, and Na_2SO_4 must be in lbs/100gal as Na_2O. At least two liquor samples and one water sample (3 total samples) are required for calibration curve parameter determination. This is true for both the manual and automated curve determination methods. In obtaining lab test values for each sample, extra care should be taken to ensure the results are as accurate as possible. The repeatability of some testing methods can be less than desirable in many instances. If this is the case, it is recommended that more than three liquor samples are used and that each one is tested multiple times. Procedures and details concerning calibration curves are located in Appendices A.6 *Manual Calibration Curve Determination* and A.7 *Automated Calibration Curve Determination*.

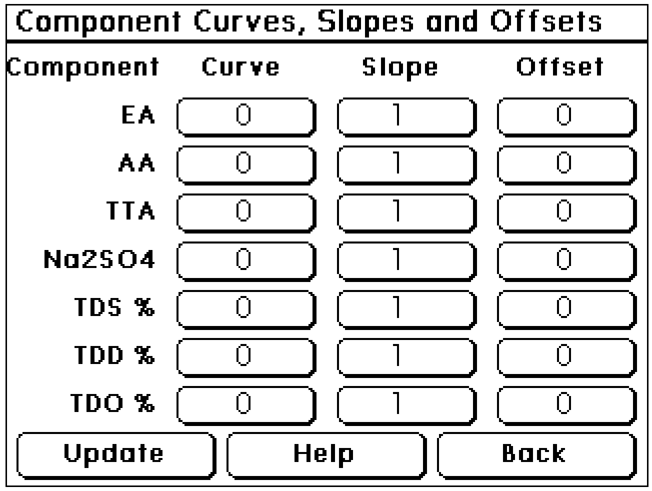


Figure 4-32. *Component Curves, Slopes and Offsets* screen with default values.

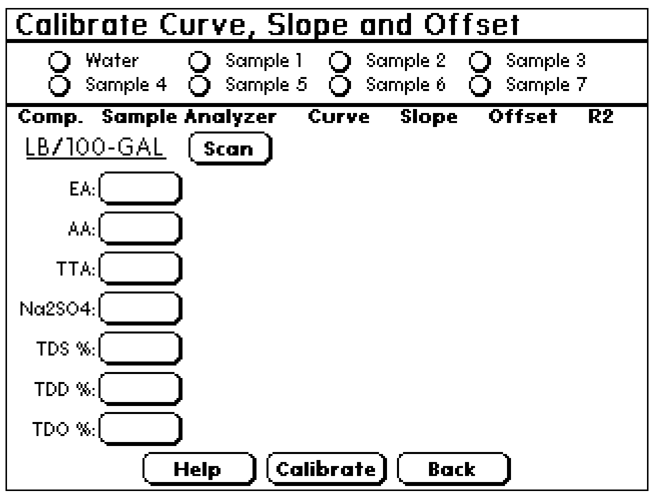


Figure 4-33. *Calibrate Curve, Slope and Offset* screen.

Every measured component for each sample point can have a scale and/or offset correction to bias the analyzer to match the manual lab tests performed onsite. If calibration curves have been generated and are in use, the bias corrections will be minimal or nonexistent. Select "Set Bias" on the *Main Menu* and then from the *Bias Adjustments* screen (Figure 4-34) select the desired sample point to bias and press "Edit". At the *Edit Bias Values* screen, enter the appropriate "Slope" and "Offset" biases and press "Update" when finished. The component (value to be measured) is listed in the left column with the corresponding slope and offset biases to the right. Default values for the *Edit Bias Values* screen are displayed in Figure 4-35. Bias adjustments (slope and/or offset) are made based on the current display units. An example of bias adjustment is given in Appendix A.8 *Bias Adjustment*.

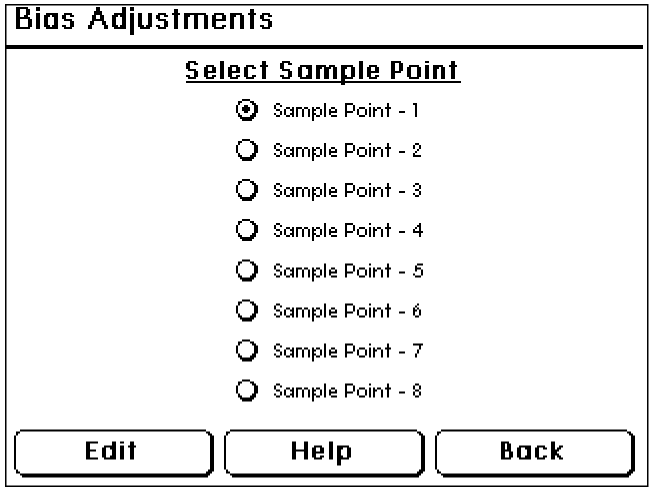


Figure 4-34. *Bias Adjustments* screen with Sample Point 1 selected.

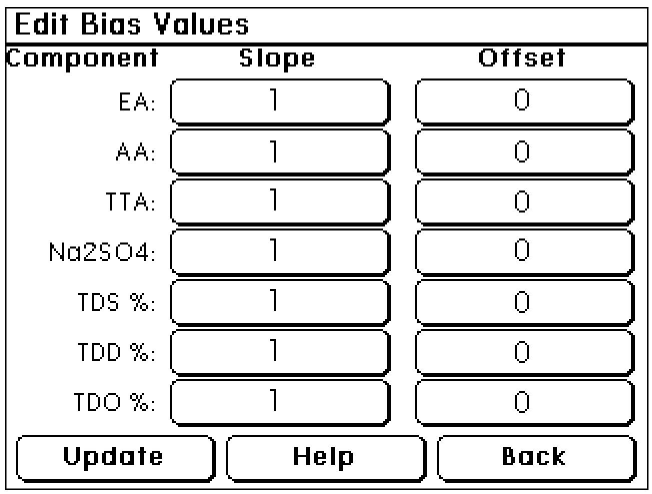


Figure 4-35. *Edit Bias Values* screen with default values.

4.5 Maintenance

The maintenance requirements for the ***DURALYZER-NIR™*** laboratory liquor analyzer are minimal both in terms of time and cost. Maintenance tasks should only be performed by qualified personnel that have read and understand this manual. There are four maintenance tasks that can be undertaken by mill personnel: **(1) Acid cleaning the sample cuvette**, **(2) Cleaning the analyzer cabinet and LCD panel**, **(3) Cleaning the thermal printer**, and **(4) Light source replacement**. Any other maintenance or repair tasks should be carried out by R. E. Hodges, LLC personnel.

Cleaning the sample cuvette is accomplished with 1.0 N or 0.1 N HCl acid (although vinegar can be used as well). The cleaning frequency will depend on the amount of use and nature of the samples being analyzed. Initially acid clean the cuvette once per day and adjust the frequency up or down if necessary. Acid is drawn into the cuvette in the same manner as water or a liquor sample and allowed to sit in the cuvette for 2 minutes. After 2 minutes, eject the acid into the waste collection bottle and flush with water.

Make certain the waste bottle has been thoroughly washed with water before ejecting the acid to avoid H_2S generation. This procedure is summarized in Appendix A.5 *Normal Operating Procedure*.

The analyzer cabinet and LCD panel should be cleaned monthly or whenever there is noticeable dust, moisture, etc. present. Simply wipe the stainless steel cabinet with a moist cloth or paper towel. A mild cleaner can be used as well. When cleaning the LCD panel, disconnect it from the analyzer. Use a soft, dry cloth for the actual screen; the LCD panel housing may be cleaned similar to the cabinet.

The presence of accumulated dust will adversely affect printer performance, possibly causing the printout to be unreadable or not to function at all. Cleaning the printer is a three part process: (1) Cleaning the thermal head, (2) Cleaning the paper holder, and (3) Cleaning the paper housing. Care must be taken when cleaning the thermal head since it is easy to damage. Use isopropyl alcohol and a soft, clean cloth. A soft cloth is sufficient for removal of dust from the paper holder. The external housing of the printer can be wiped off with a moist cloth.

The typical lifespan of the QTH light source should be approximately one year. Before the bulb completely expires, there are usually signs the end of the lifespan is approaching. A drop in bulb intensity combined with erratic measurements will be the most noteworthy characteristics. A good rule of thumb is to order a replacement QTH NIR light source after 11 months of operation and install the new bulb after 12 months.

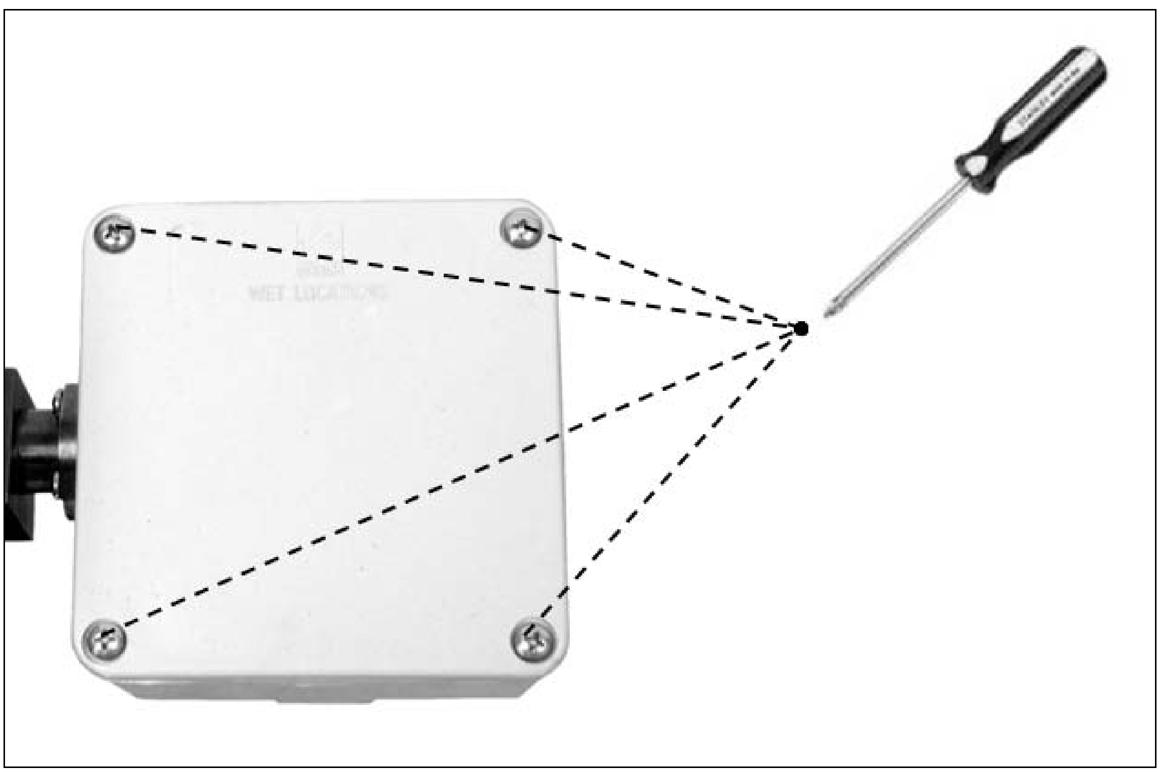


Figure 4-36. NIR light source enclosure.

To replace the NIR light source, power off the analyzer and remove the light source enclosure lid with a Phillips head screwdriver (Figure 4-36). Use an Allen wrench to loosen the light source holder set screw, unplug and then remove the old bulb (Figure 4-37, **WARNING**: *Bulb and light source holder will be extremely hot if the old bulb was not completely burned out*). Insert the new bulb into the holder, tighten the set screw, power on the analyzer, and replace the cover. Allow the new bulb to stabilize for two hours before using the analyzer. Utilize the "New Bulb" function on the Main Menu screen to test for detector saturation (see Section 4.4.4 *Informative Submenus*) and adjust the bulb position if necessary. For further details concerning light source replacement refer to Appendix A.10 *NIR Light Source Replacement*.

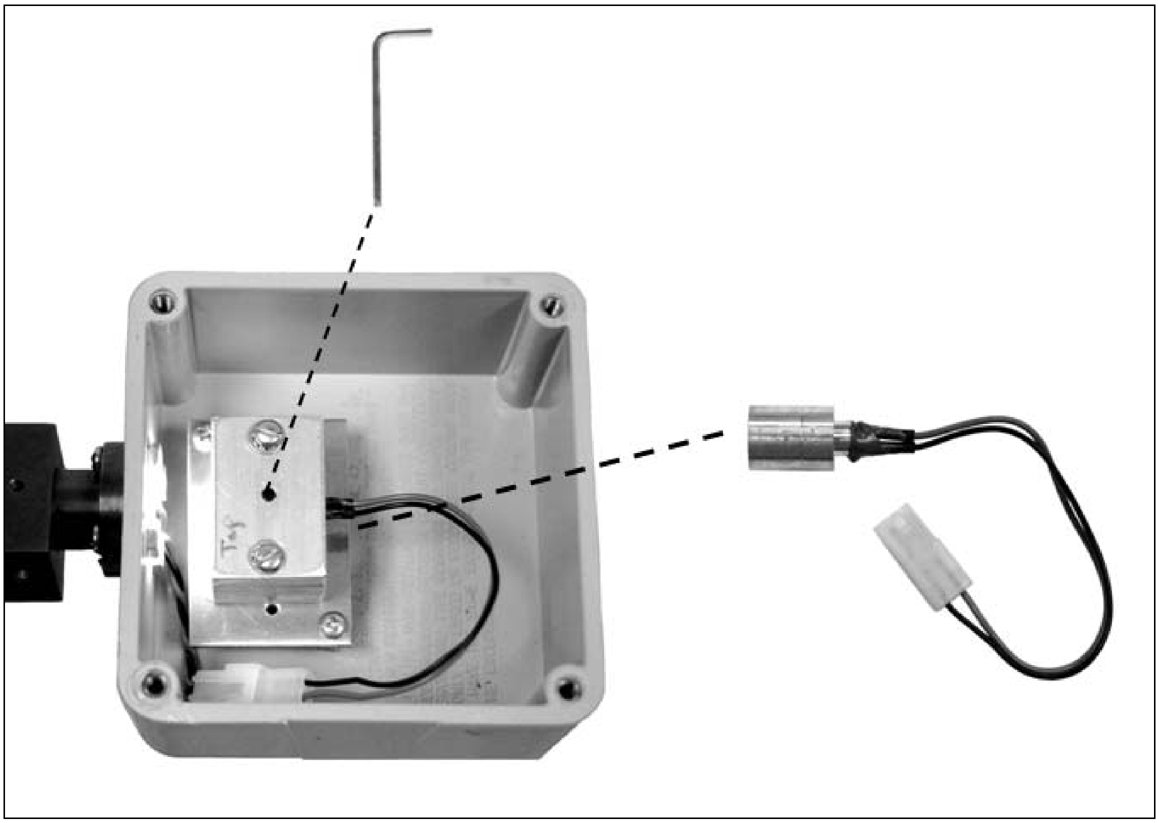


Figure 4-37. NIR light source removal.

Certain other unscheduled maintenance tasks may occur over the operational lifetime of the analyzer. These will be relatively simple and covered in Appendix A.12 *Troubleshooting*. Any significant maintenance or major analyzer component replacement would have to be performed by R. E. Hodges, LLC. Any repair or maintenance performed on the analyzer that has not been explicitly discussed in this manual will void the warranty.

Appendix

Appendix A.1 Dimensions and Additional Images

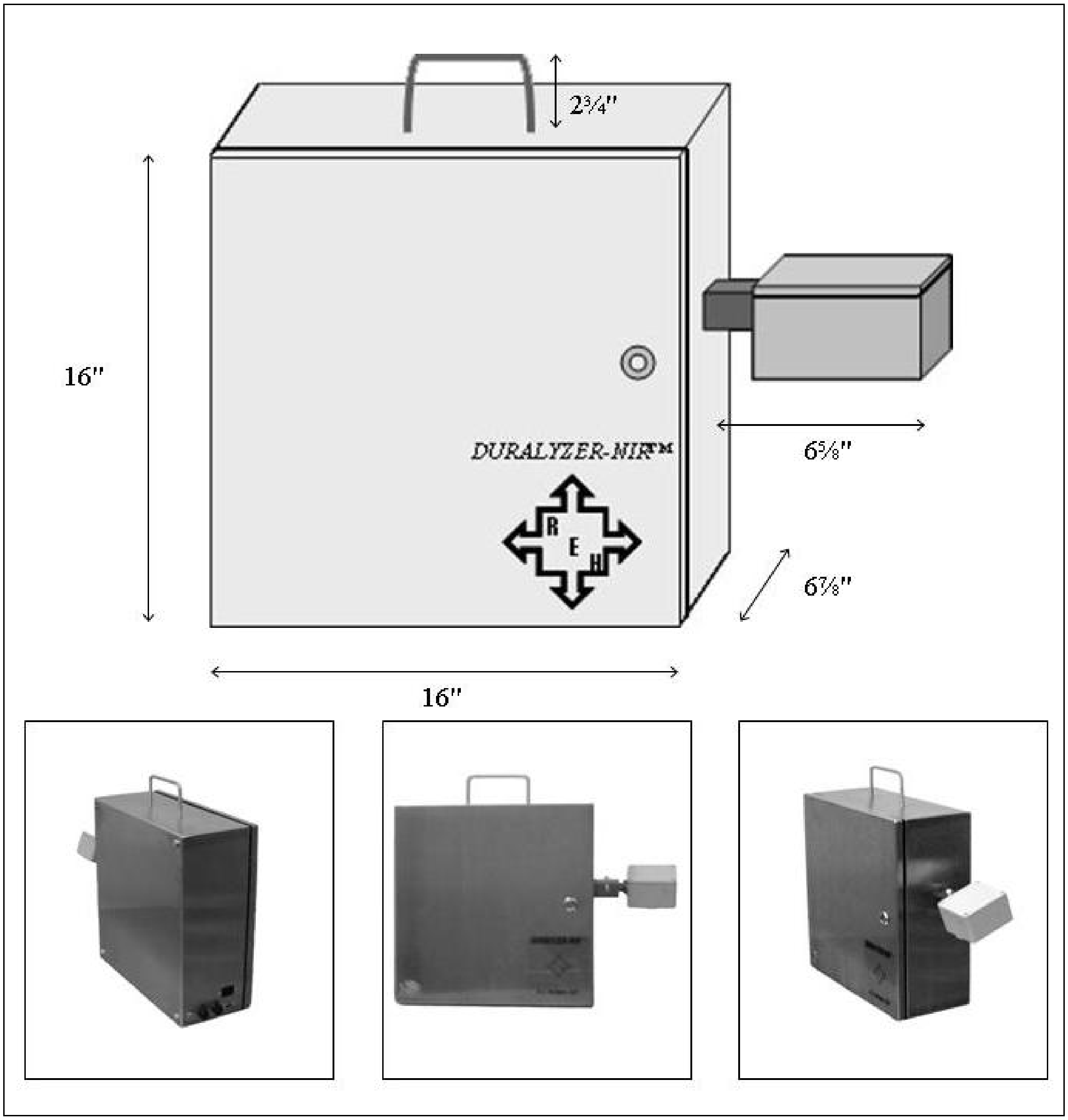


Figure A.1-1. ***DURALYZER-NIR™*** laboratory liquor analyzer dimensions.

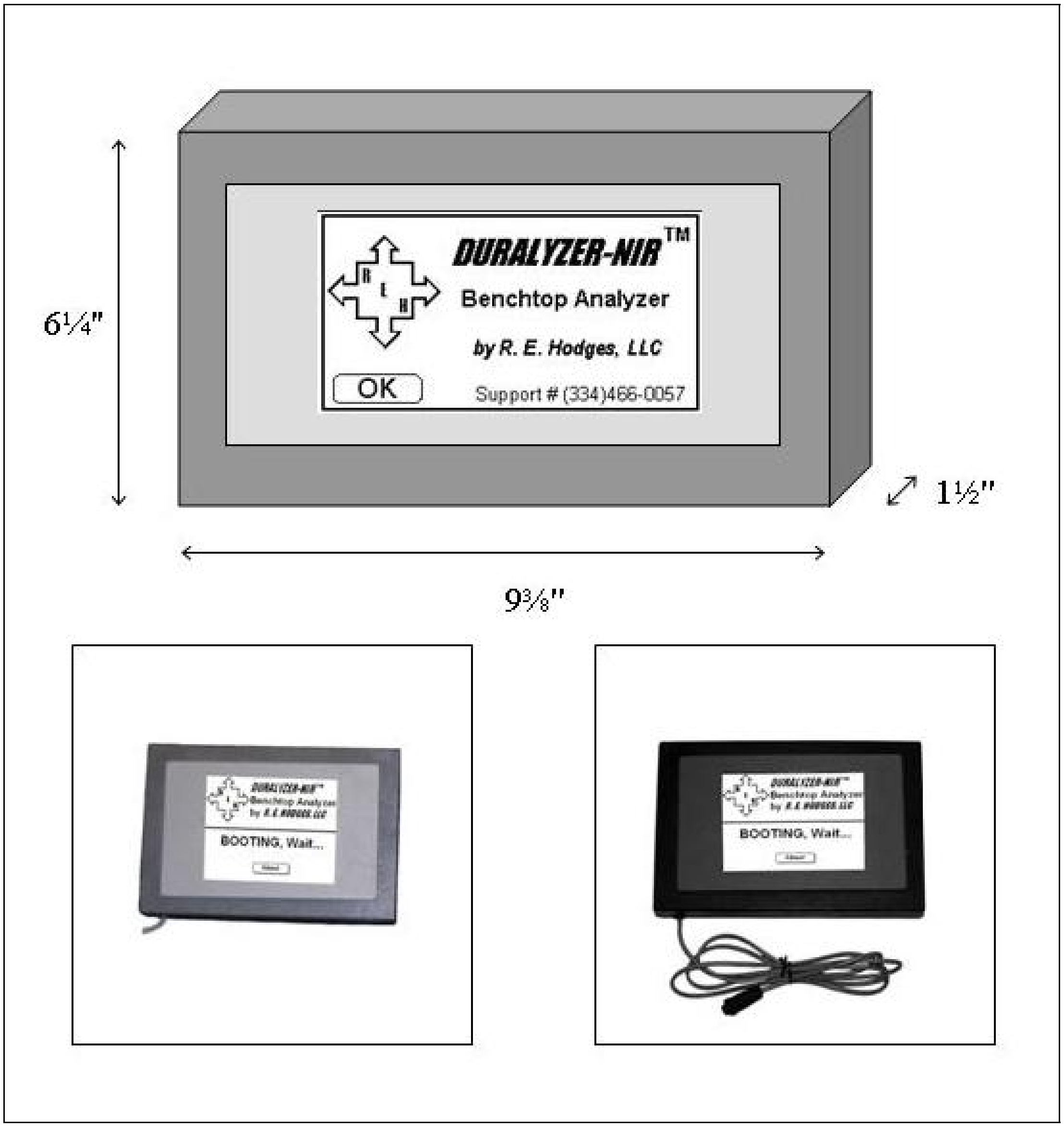


Figure A.1-2. LCD panel dimensions.

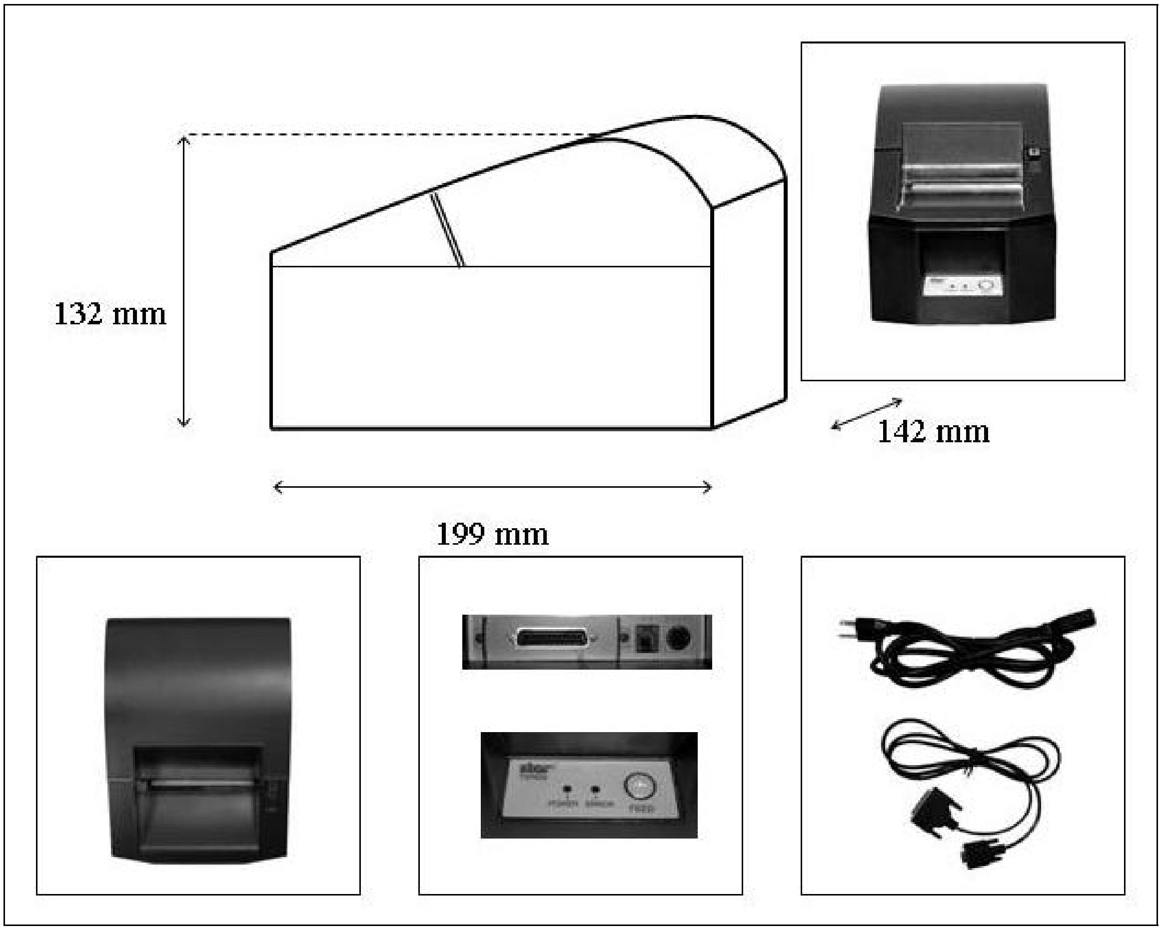


Figure A.1-3. Printer dimensions.

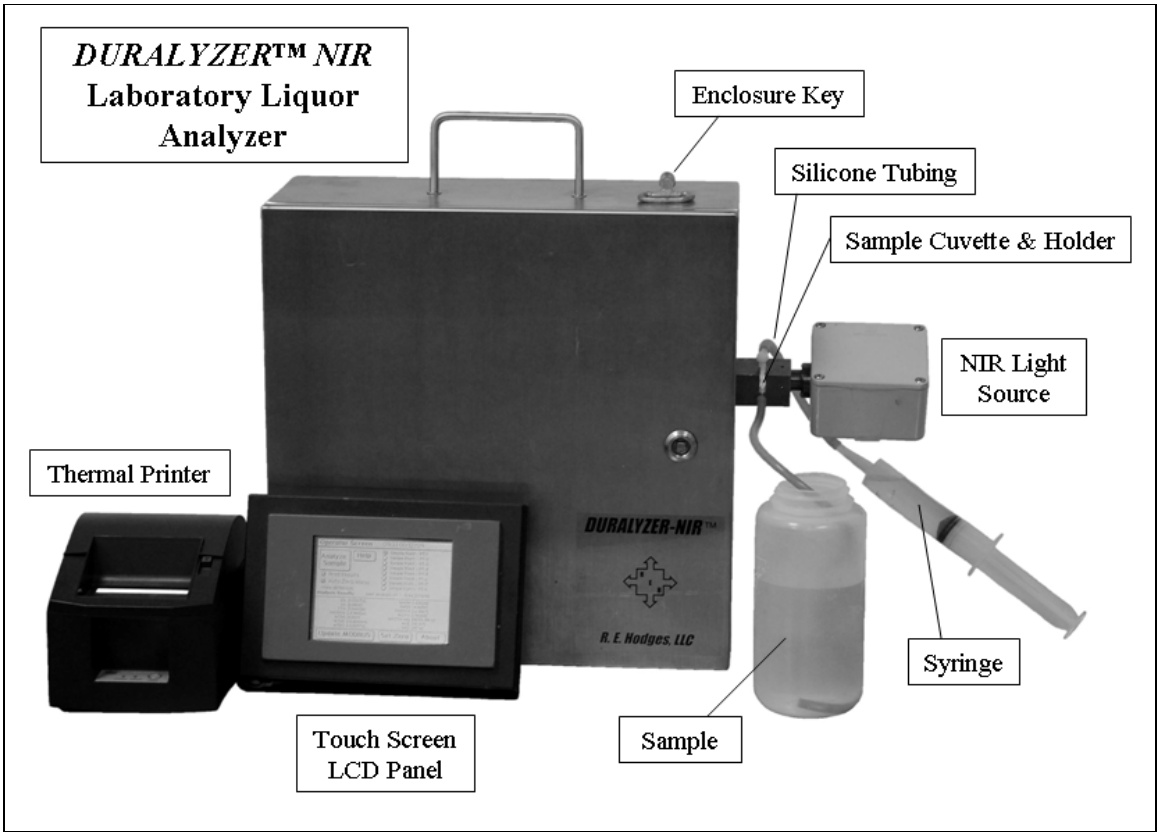


Figure A.1-4. System setup.

Figure A.1-5. NIR Quartz-Tungsten-Halogen (QTH) light source.

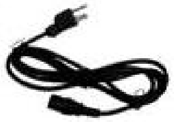
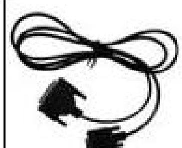
	Analyzer power cable (6 ft)		Printer
	Sample cuvette (2)		Printer AC adapter w/ 5 ft cable
	Enclosure key		Printer power cable (6 ft)
	LCD touch screen panel w/ 5½ ft cord		Printer serial interface cable (6 ft)
	60 ml rubber pipetting bulb		Silicone tubing (4 ft)

Figure A.1-6. Accessories and cables.

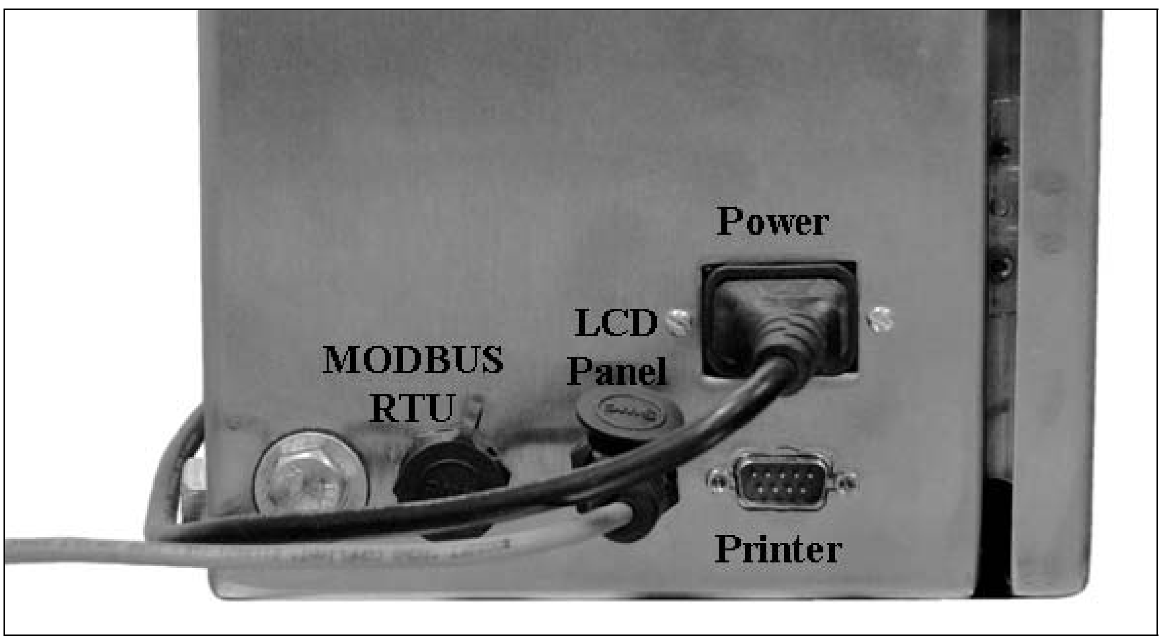


Figure A.1-7. Analyzer external connection ports.

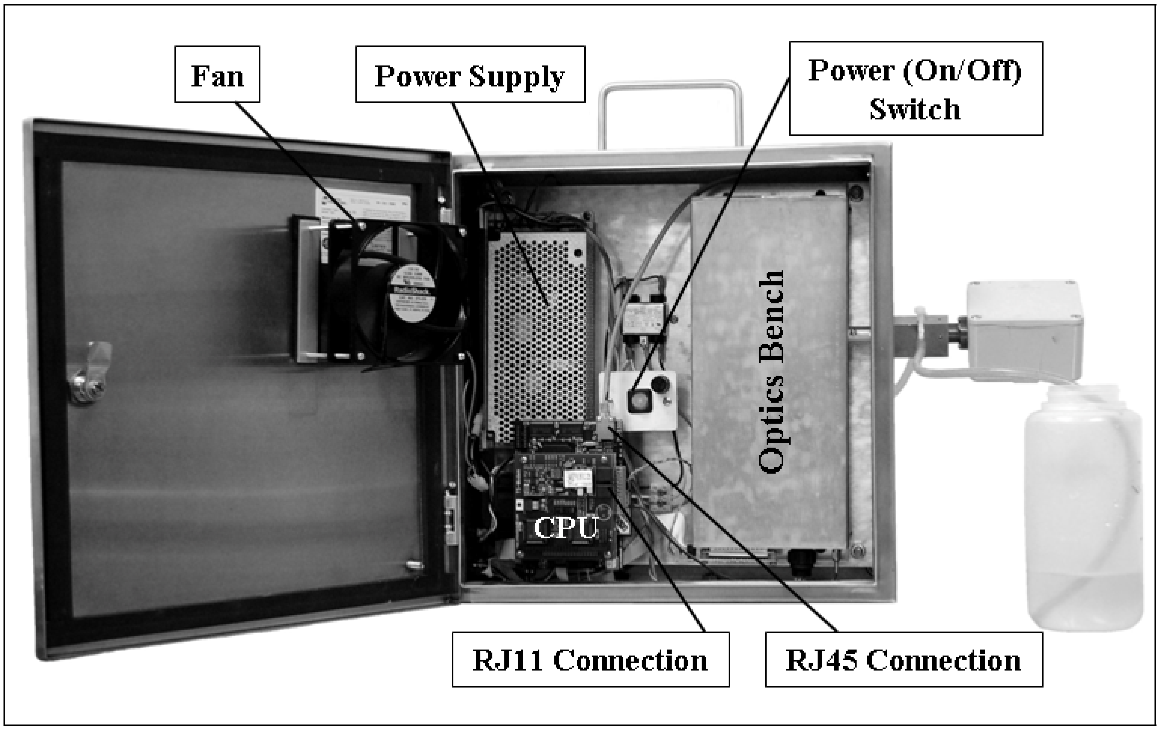


Figure A.1-8. Analyzer internal components.

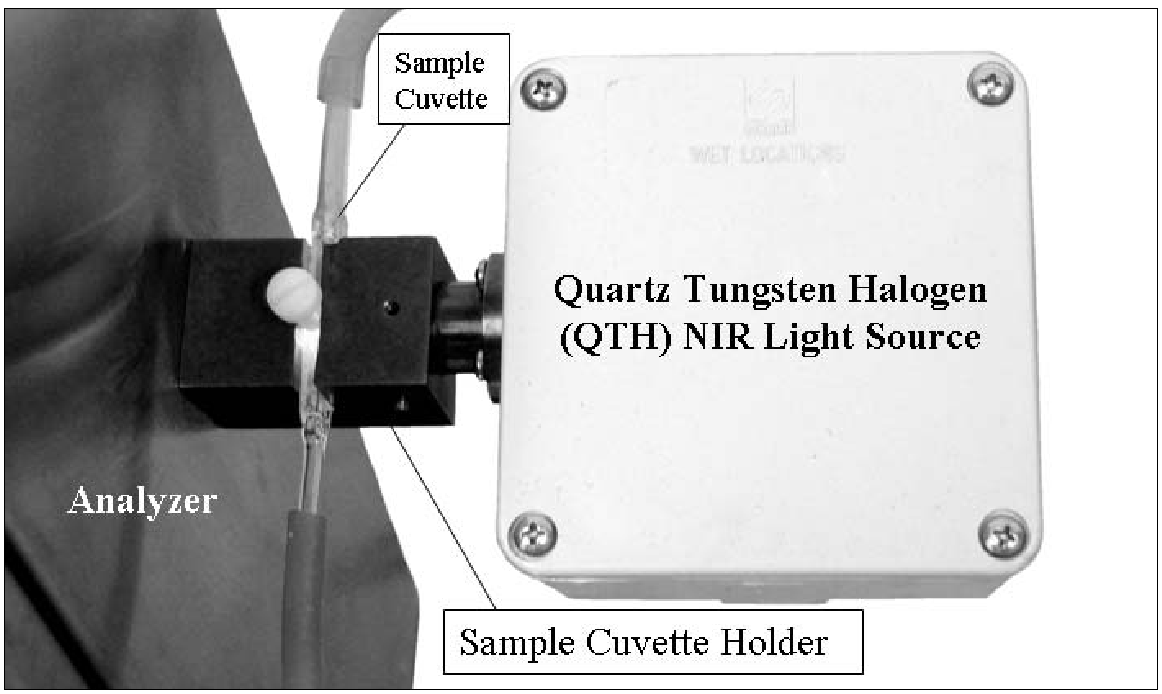


Figure A.1-9. Sample cuvette and light source enclosure.

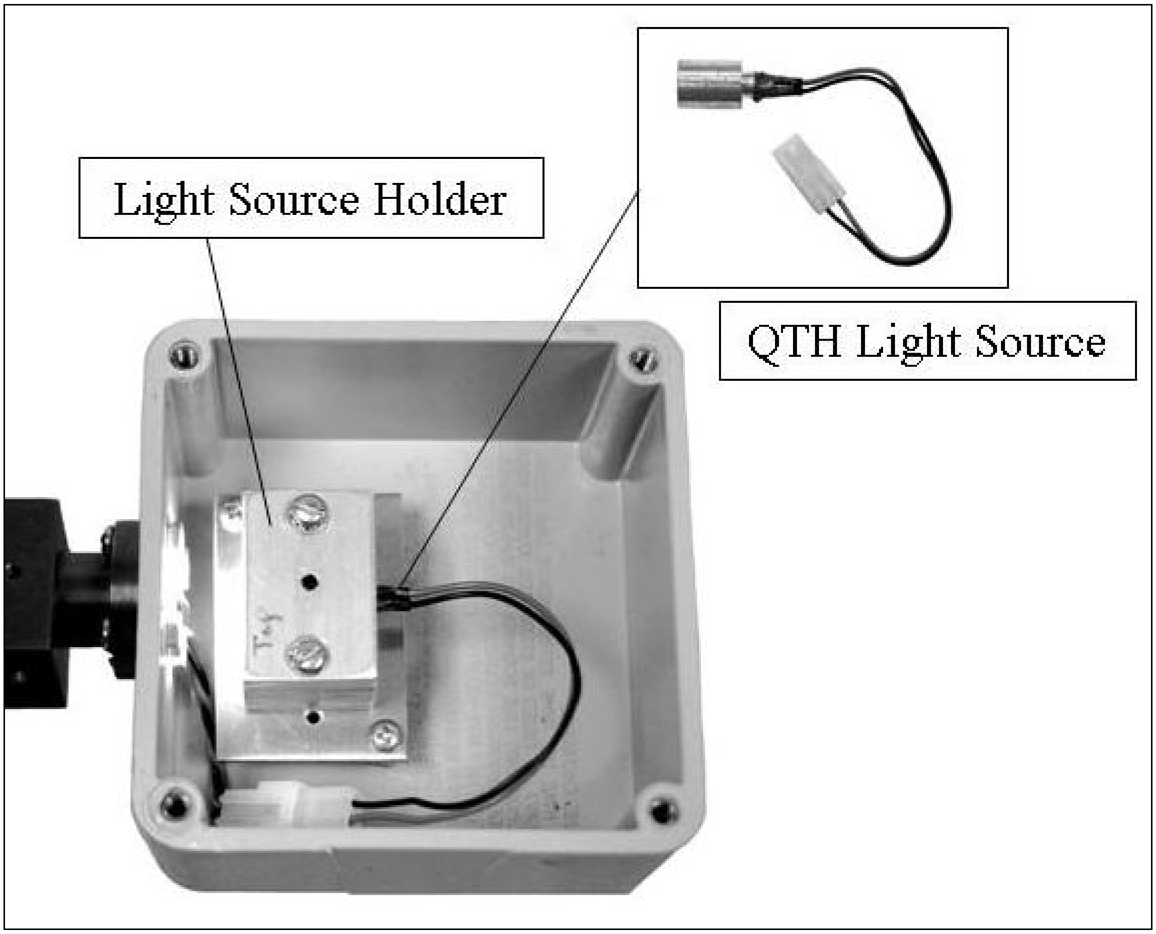


Figure A.1-10. NIR light source enclosure.

Figure A.1-11. Analyzer side view.

Appendix A.2 Nomenclature

AA	Active Alkali
CE	Causticizing Efficiency
DCS	Digital Control System
EA	Effective Alkali
KCl	Potassium Chloride
LCD	Liquid Crystal Display
MODBUS	Modicon Communications Bus
Na_2CO_3	Sodium Carbonate
Na_2S	Sodium Sulfide
$Na_2S_2O_3$	Sodium Thiosulfate
Na_2SO_3	Sodium Sulfite
Na_2SO_4	Sodium Sulfate
NaCl	Sodium Chloride
NaOH	Sodium Hydroxide
NEMA	National Electrical Manufacturers Association
NIR	Near Infrared
QTH	Quartz Tungsten Halogen
RAA	Residual Active Alkali
RE	Reduction Efficiency
REA	Residual Effective Alkali
REH	R. E. Hodges, LLC
RTU	Remote Terminal Unit
SCAN	Scandinavian Pulp, Paper and Board Testing Committee
SS	Stainless Steel
TAPPI	Technical Association of the Pulp and Paper Industry
TCP	Transmission Control Protocol
TDD	Total Dissolved Dead Load
TDS	Total Dissolved Solids
TDO	Total Dissolved Organics
TEC	Thermoelectric Cooler
TTA	Total Titratable Alkali
VAC	Volts Alternating Current
A	Second Order Calibration Curve Parameter
B	First Order Calibration Curve Parameter
C	Offset Calibration Curve Parameter
V	Volume of Sample
S_0	Bias Offset Parameter
S_1	Bias Slope Parameter
m	Mass of sample
x_b	Bias Adjusted Analyzer Value
x_{cc}	Curve Corrected Analyzer Value
x_{nz}	Analyzer Water Zeroed Value Prior to Curve Correction
x_{ra}	Raw Analyzer Value Prior to Water Zero and Curve Correction
ρ	Density (m/V)

Appendix A.3 Useful Equations

EA:	$EA = NaOH + \frac{1}{2} Na_2S$	(as Na_2O)
AA:	$AA = NaOH + Na_2S$	(as Na_2O)
TTA:	$TTA = NaOH + Na_2S + Na_2CO_3$	(as Na_2O)
NaOH:	$NaOH = 2EA - AA$	(as Na_2O)
Na_2S:	$Na_2S = 2(AA - EA)$	(as Na_2O)
Na_2CO_3:	$Na_2CO_3 = TTA - AA$	(as Na_2O)
%Sulfidity (TTA-basis):	$\%Sulfidity = \frac{Na_2S}{TTA} \cdot 100\%$	(Na_2O basis)
%Sulfidity (AA-basis):	$\%Sulfidity = \frac{Na_2S}{AA} \cdot 100\%$	(Na_2O basis)
%Causticity:	$\%Causticity = \frac{NaOH}{AA} \cdot 100\%$	(Na_2O basis)
%RE:	$\%RE = \frac{Na_2S}{Na_2S + Na_2SO_4} \cdot 100\%$	(Na_2O basis)
%CE:	$\%CE = \frac{NaOH}{NaOH + Na_2CO_3} \cdot 100\%$	(Na_2O basis)

%TDS: $$\%TDS = \frac{mass\ of\ solids}{mass\ of\ solution} \cdot 100\% = (\rho - 1) \cdot 100\%$$ (ρ is in g/cm^3)

%TDD:

$$\%TDD = \frac{mass\ of\ (Na_2SO_4 + Na_2SO_3 + Na_2S_2O_3 + KCl + NaCl)}{mass\ of\ solution} \cdot 100\%$$

$$= \%TDS - \left[\frac{(1.291 * NaOH) + (1.259 * Na_2S) + (1.710 * Na_2CO_3)}{\rho * 1000 * 0.834} \right]$$

(*NaOH*, *Na_2S*, and *Na_2CO_3* in lb/100gal as Na_2O, ρ is in g/cm^3 in the bottom equation)

Appendix A.4 Setup Cautions

When using (or storing) the ***DURALYZER-NIR™*** laboratory liquor analyzer, be sensible with regard to the setup location. Care should be taken to **avoid the following conditions**:

- High humidity
- High temperatures
- Volatile materials
- Areas susceptible to wetness or splashing
- Vibration
- Excessive dust levels
- Open flames
- Abrupt changes in humidity and/or temperature
- Direct exposure to strong acids or acid vapors

Appendix A.5 Normal Operating Procedure

Supplies Required:

1. 1.0 N or 0.1 N HCl acid for cleaning sample cuvette.
2. 250 mL (or larger) Nalgene® sample bottles.
3. Sample waste collection bottle (1.0 L Nalgene® sample bottle).
4. Container with room temperature potable water (for zeroing the analyzer and flushing the sample cell).

Cleaning Procedure:

1. Acid wash the sample cuvette with 1.0 or 0.1 N HCl **once per day** (the frequency of cleaning may be relaxed depending on scaling conditions and the number of samples being analyzed each day). Draw in the acid and let sit for approximately 2 minutes.
2. Eject the acid from the sample cuvette and flush with water. If samples are not going to be analyzed immediately, draw water into the cuvette and let remain until samples are to be tested. Otherwise, proceed with sample testing procedure.
3. When ejecting acid into the waste collection bottle, **make certain the bottle has been thoroughly washed with water to avoid H_2S generation**.

"Set Zero" Procedure:

1. Fill a sample bottle/container with water.
2. Fully compress the pipetting bulb and then insert the tubing into the water. Draw the water into the cuvette by slowly releasing the bulb. Allow the bulb to hang freely when uncompressed (fully expanded).
3. Select (touch) "Set Zero" on the *Operator Screen*. After a 2 minute count down, the analyzer will scan (acquire spectrum) the water. The "Zero" has now been set. (The baseline has been set and the internal spectrometer optical alignment has been checked.)
4. Eject the water into the sample waste bottle by holding the bulb in an upright position and compressing (squeezing) several times to eject all the water. The tubing should be held or secured when doing this to avoid the possibility of uncontrolled spray. Note: if samples are not to be analyzed immediately, leave the water in the cuvette (i.e., do not eject the water until ready to test samples). The "Zero" should be set at least once per day and every time following an acid cleaning or after powering on the analyzer.

Sample Testing Procedure:

1. Collect all samples to be tested from the field. Samples need to be large enough to allow for at least a 200 mL viable sample after any settling or filtering (see below).

2. *For slurry samples (such as the No. 1 causticizer) and raw green liquor samples*, allow ample settling time before transferring the settled (clear) liquor to a new (clean) 250 mL sample bottle.
3. *For black liquor samples containing chip and/or fiber debris*, pour the sample through a 100 mesh strainer into a new (clean) 250 mL sample bottle. This will remove all of the debris that could possibly interfere with the analysis.
4. *Optional*: Samples can be capped and placed in a water bath to bring them closer to room temperature. Place the capped samples in a large container (such as a pot). Put the large container in a sink and fill with tap water. Keep the water running and allow the water to overflow from the container for at least 2 minutes before removing the sample containers from the water bath and proceed with testing. Cooling the samples can stability and repeatability in the analysis results. As a general rule of thumb, each sample point should consistently be tested in the same 10 °C window for optimal results.
5. If the "Auto Zero" function is being used, uncheck the corresponding box on the *Operator Screen* (to disable the function) until sample testing is complete.
6. Select (touch) the circle next to corresponding sample point for testing on the *Operator Screen*. (If water is still in the cuvette, eject into the sample waste bottle by holding the bulb in an upright position and compressing (squeezing) several times to eject all the water. The tubing should be held or secured when doing this to avoid the possibility of uncontrolled spray.)
7. Fully compress the bulb and then place the sample pickup tube in the sample bottle corresponding to the sample point selected in step 6.
8. With the tube in the sample bottle, slowly release the bulb until completely expanded and then allow it to hang freely. Make certain that the end of the sample pickup tube is not in contact with the bottom of the bottle in a manner that will interfere with suction of the sample. *For slurry samples*, the very bottom should be avoided if a detectable amount of residual lime mud has settled. *For black liquor samples*, ensure the pickup tube end is below the soap layer of liquor to minimize the amount of soaps and extractives pulled into the cuvette.
9. Select "Analyze Sample" on the *Operator Screen*. The sample will be analyzed after the delay time (default is 120 seconds). The delay is necessary to ensure the sample has stabilized in the cuvette (an in some cases will need to be increased beyond the default value of 120 seconds). When the analysis is complete, the *Operator Screen* will update, displaying the current results.
10. If the MODBUS communication interface is being used, select "Update MODBUS" on the *Operator Screen* to send the analysis results to the appropriate registers.
11. For a hard copy printout of the analysis results (via the thermal printer), check the "Print Results" box.

12. Place the pickup tubing in the sample waste collection bottle and eject the liquor by holding the bulb in an upright position and compressing (squeezing) several times to eject all the liquor. The tubing should be held or secured when doing this to avoid the possibility of uncontrolled spray. Thoroughly flush the sample cuvette with water and eject into the waste bottle.
13. If analyzing (testing) more samples, repeat steps 6-12.
14. When the testing session is complete (all samples acquired from the field have been analyzed), fill a clean container with water and draw it into the sample cuvette. Allow the water to remain in the cuvette until the next series of samples are to be tested.

Appendix A.6 Manual Calibration Curve Determination

Background:

The ***DURALYZER-NIR™*** laboratory liquor analyzer has seven standard calibration (predictive) models for analyzing green, white, and black liquor samples. The calibration models have been developed through a combination of signal processing steps and mathematical regression techniques. Models are tuned by utilizing a calibration curve. Employing a calibration curve allows the individual component (EA, AA, etc.) models to be adjusted to match the lab test method (results) in a least squares sense. The seven models are as follows:

1. EA (REA for black liquor)
2. AA (RAA for black liquor)
3. TTA
4. Na_2SO_4
5. % TDS
6. % TDD
7. % TDO

Certain measurements may not be required for a given application of the analyzer. In which case, determining a calibration curve for that model would be unnecessary. For example, if there is not an interest in Reduction Efficiency then there is not a need for a sodium sulfate calibration curve. Likewise, %TDO (total dissolved organics) would only have meaning for black liquor samples.

For *manual* determination, each calibration curve will be represented by the second order polynomial given by Equation A.6-1.

$$x_{cc} = Ax_{nz}^2 + Bx_{nz} \qquad \text{(A.6-1)}$$

Constant curve parameters, A and B, are determined by the manual calibration curve procedure, x_{cc} is the "curve corrected" measurement value, and x_{nz} is the "water-zeroed" analyzer value prior to any curve correction or bias adjustment. Both x_{cc} and x_{nz} must be in lb/100gal as Na_2O. The base units for the EA, AA, TTA, and Na_2SO_4 models are lb/100gal. There is not a "C" term in Equation A.6-1 because that has already been incorporated into x_{cc} by setting the water zero. Each calibration model will have its own set of A and B curve parameters.

Samples:

Follow the *Normal Operating Procedure* (Appendix A.5) for analyzing the samples. At a minimum, two liquor samples and one water sample (3 total samples) are required for calibration curve parameter determination. Due to setting the water zero, the analyzer and lab measurement values for water will be "0.0" and "0.0". If, the current lab testing method in place produces less than desirable repeatability, more than two liquor samples will be required. It is important to note that all samples need to be tested using the same method and by the same person. This eliminates potential of conflicting biases being

introduced into the lab values and ultimately will yield a less than optimal calibration curve. Special care should be taken when performing the lab tests to ensure the highest possible precision in the measurement values.

For example, past experience has shown that using an autotitrator for the white liquor "ABC" test and manual titration for the green liquor "ABC" test is not a good idea. If this is the standard treatment at a mill, run the green and white liquor tests for curve parameter determination on the autotitrator. After the calibration curves are set and in use, go back and run manual tests on the green liquor and make any adjustments by using the "Set Bias" function on the LCD panel *Main Menu*. Details on setting biases can be found in Section 4.4.5 and Appendix A.8.

For EA and AA, a white liquor and a green liquor sample is recommended to determine the curve parameters. A weak wash sample and/or a black liquor sample can be used as well. The important idea is to have samples that give values across a wide range and between the upper and lower values (i.e., green liquor EA and AA falls between water and white liquor). Black liquor with very low REA or high organic and soap content is not ideal for curve parameter determination. In this case, any black liquor measurement adjustments should be made through the "Set Bias" function. If there is only a white liquor sample available, then a white liquor sample may be diluted to form a second liquor sample. A one part white liquor to one part water dilution ratio would work fine and yield an EA and AA value roughly half of the undiluted sample. The dilute sample must still be tested to obtain a precise lab measurement value (do not assume it is exactly half the undiluted value).

The TTA calibration curve can be determined from green liquor, weak wash, and white liquor samples. Other combinations are possible as well, such as weak wash and white liquor or white liquor and diluted white liquor (see above paragraph for comments on dilution).

Reduction efficiency (RE%) computation requires the measurement of sodium sulfate. Green liquor samples are used to determine the Na_2SO_4 curve parameters. Obtain a single green liquor sample in an amount suitable to divide into three samples of equal volume. Add approximately 5 g/L and 10 g/L of $Na_2SO_{4(s)}$ to samples one and two respectively. Do not add any to the third sample. Allow the sodium sulfate added to samples one and two to completely dissolve (this will take more time the cooler the sample is). Next, test all three samples using the standard laboratory test procedure for sodium sulfate in green liquor.

Determination of the %TDS calibration curve parameters can be carried out using white, green and black liquors. A convenient relationship for calculating %TDS in white and green liquor is depicted in Equation A.6-2.

$$\%TDS = (\rho - 1.000) \cdot 100\% \qquad \text{(A.6-2)}$$

The density (ρ) is in g/cm^3 and can be measured by a handheld density meter or by a gravimetric method. At room temperature, measure out a precise volume of sample (V) in mL then determine the mass (m) in grams. The black liquor %TDS is usually measured by a moisture analyzer or by oven drying.

Only green and white liquor samples are used to calculate the %TDD calibration curve. Lab values for %TDD can be calculated from the following equation if the EA, AA, TTA, ρ, and %TDS values are known.

$$\%TDD = \%TDS - \left[\frac{(1.291 * NaOH) + (1.259 * Na_2S) + (1.710 * Na_2CO_3)}{\rho * 1000 * 0.834} \right] \qquad \text{(A.6-3)}$$

Density (ρ) is in g/cm^3, whereas NaOH, Na_2S, and Na_2CO_3 are in lb/100gal as Na_2O.

Determining the %TDO calibration curve requires black liquor samples with a known %TDS. A precisely measured mass of black liquor is dried and burned in a furnace to measure the total dissolved organics. The %TDO is computed by Equation A.6-4.

$$\%TDO = \%TDS - \left(\frac{remaining\ mass\ of\ burned\ sample}{mass\ of\ wet\ sample} \right) \cdot 100 \qquad \text{(A.6-4)}$$

Procedure:

1. Make sure the analyzer has been **powered on for a minimum of 2 hours (12+ hours recommended)** to allow for the analyzer to reach thermal equilibrium and stabilize. Confirm that "Curve", "Slope", and "Offset" on the *Component Curves, Slopes and Offsets* screen are set to "0", "1", and "0" respectively for each component model requiring a calibration curve. Do the same on the *Edit Bias Values* screen, setting the "Slope" and "Offset" to "1" and "0" respectively (this must be done for each sample point). Accessing these screens was detailed in Section 4.4.5 *Model Tuning Submenus*. It is also helpful to set the display units to lb/100gal for EA, AA, TTA, and Na_2SO_4 (refer to Section 4.4.3 *Configuration Submenus*).
2. Collect all liquor samples that will be required for determining the calibration curve parameters.
3. Perform the necessary laboratory tests on the collected samples. The tests will yield the "LAB" values.
4. After lab testing is complete, set the water zero. Refer to Section 4.3 *Normal Operation* and Appendix A.5 *Normal Operating Procedure*.
5. Test the collected samples on the ***DURALYZER-NIR™*** laboratory liquor analyzer following the procedures set forth in Section 4.3 and Appendix A.5. After each sample has been analyzed, record the values shown in the "Analysis Results" section of the *Operator Screen*. These will be the analyzer or "NIR" values. It is highly recommended to have the "Print Results" box checked on the *Operator Screen* and the printer connected prior to analyzing the samples.

6. After all collected samples have been lab tested and analyzer tested, the mathematical determination of the calibration curve parameters is performed. This requires the use of spreadsheet software such as Microsoft® Excel.
7. On the spreadsheet, make three column headings for the sample, the analyzer measurement value and the lab measurement value. If any of the measured values are in units other than lb/100gal, make new columns with the values in lb/100gal. The first sample will always be "water" and its corresponding analyzer and lab values will always be "0.00" (due to setting the water zero). This is arrangement is done for each component.
8. Construct a scatter plot with the analyzer values (lb/100gal) being the "X" data and the lab values (lb/100gal) being the "Y" data. Fit the data with a 2^{nd} order polynomial trendline and set the intercept to zero. The resulting trendline equation is the calibration curve in the form of Equation A.6-1. In some cases, it may be preferable to fit the data with a linear trendline (and the "A" term in Equation A.6-1 will be zero).
9. Repeat step 8 for all components requiring a calibration curve.
10. After determining the calibration curve equations (parameters), enter the corresponding values into the *Component Curves, Slopes and Offsets* screen. Referring to Equation A.6-1, "A" is the "Curve" value, "B" is the "Slope" value, and "0.0" is the "Offset" value. When done, select "Update". The calibration curves are now set and will be in use the next time a sample is analyzed.

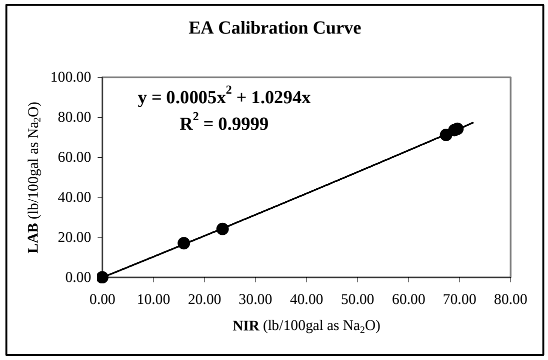
Two examples are presented to further highlight and explain the procedure and concepts presented above.

<u>Example A.6-1</u>:

In the following example, the ***DURALYZER-NIR™*** laboratory liquor analyzer is operated in causticizing area of the mill. The following samples will be analyzed on a regular basis: #1 causticizer, #3 causticizer, white liquor, weak wash, and green liquor. Measurements of particular interest are EA, AA, TTA, %Sulfidity, %CE, and %TDS. Calibration curves for EA, AA, TTA, and %TDS need to be determined and implemented. Figure A.6-1 illustrates the calculation of the curve parameters based on data provided in lb/100gal as Na_2O. A 2^{nd} order polynomial fit is utilized for the EA, AA, and TTA, whereas a linear fit is applied to the %TDS data. The calibration curve equations are clearly highlighted for each calibration model.

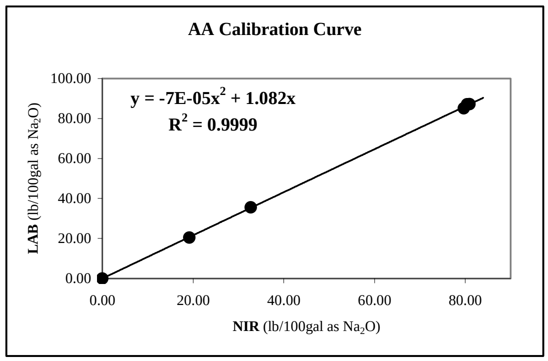
SAMPLE	EA_NIR (lb/100gal as Na_2O)	EA_LAB (lb/100gal as Na_2O)
Water	0.00	0.00
#1 Causticizer	67.36	71.11
#3 Causticizer	69.02	73.50
White Liquor	69.61	74.14
Weak Wash	15.98	17.03
Green Liquor	23.57	24.21

$$x_{cc,EA} = 0.0005\, x_{nz,EA}^2 + 1.0294\, x_{nz,EA}$$



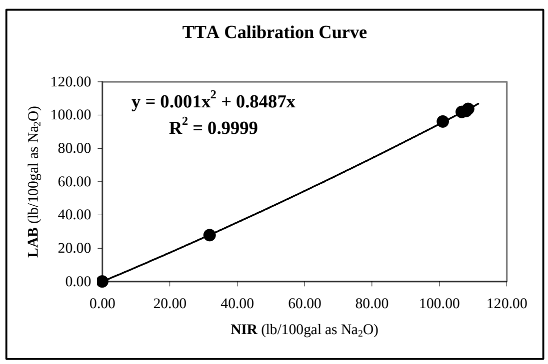
SAMPLE	AA_NIR (lb/100gal as Na_2O)	AA_LAB (lb/100gal as Na_2O)
Water	0.00	0.00
#1 Causticizer	79.70	85.09
#3 Causticizer	80.40	87.07
White Liquor	80.94	87.24
Weak Wash	19.18	20.47
Green Liquor	32.73	35.56

$$x_{cc,AA} = -0.00007\, x_{nz,AA}^2 + 1.082\, x_{nz,AA}$$



SAMPLE	TTA_NIR (lb/100gal as Na_2O)	TTA_LAB (lb/100gal as Na_2O)
Water	0.00	0.00
#1 Causticizer	108.52	103.75
#3 Causticizer	107.98	102.40
White Liquor	106.69	101.91
Weak Wash	31.83	27.88
Green Liquor	101.01	96.12

$$x_{cc,TTA} = 0.001\, x_{nz,TTA}^2 + 0.8487\, x_{nz,TTA}$$



SAMPLE	TDS_NIR (%)	TDS_LAB (%)
Water	0.00	0.00
#1 Causticizer	20.48	17.98
#3 Causticizer	19.89	17.24
White Liquor	19.51	16.94
Weak Wash	N/A	N/A
Green Liquor	22.94	20.39

$$x_{cc,\%TDS} = 0.000 x_{nz,\%TDS}^2 + 0.8766 x_{nz,\%TDS}$$

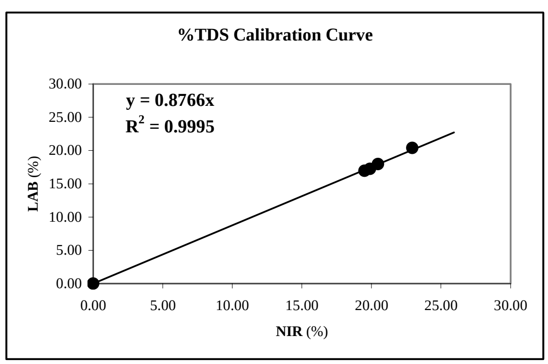


Figure A.6-1. Calibration curves for Example A.6-1.

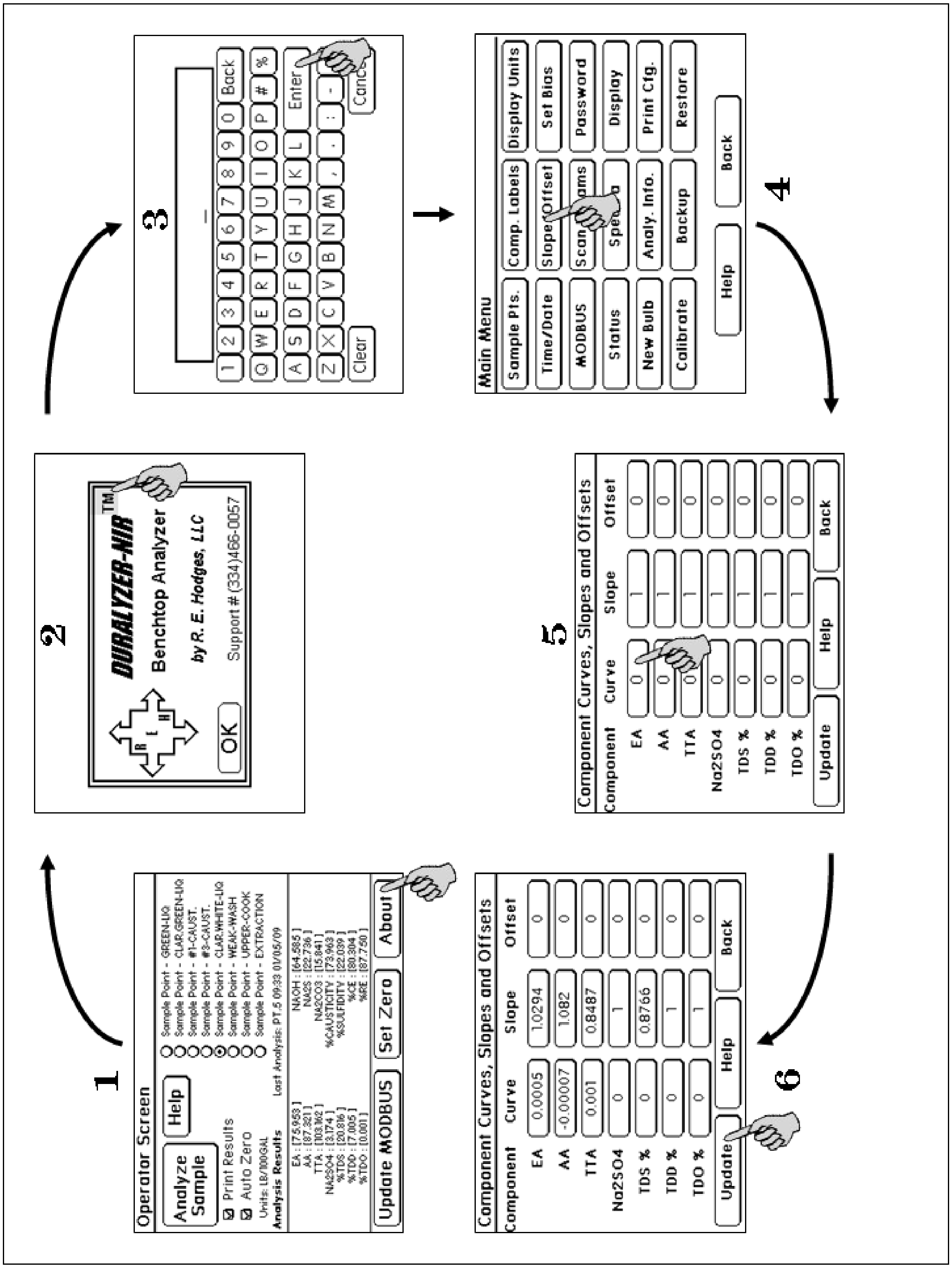


Figure A.6-2. Menu sequence for manual entry of calibration curve parameters.

Figure A.6-2 illustrates the menu navigation for entering the calibration curve parameters. Starting from the *Operator Screen* (1), "About" is selected and screen (2)

appears. Pressing "TM" brings up screen (3) to enter the password which allows access to the *Main Menu* (4). From the *Main Menu* (4), "Slope/Offset" is selected and the calibration parameters are entered on the *Component Curves, Slopes and Offsets* screen (5). Screen (6) shows the parameters determined in *Example A.6-1*. Once the curve parameters have been entered, select "Update" on screen (6) for the changes to take effect.

Table A.6-1. Comparison of analyzer values with lab values.

SAMPLE	EA*			AA*		
	NIR	CC	LAB	NIR	CC	LAB
Water	0.00	0.00	0.00	0.00	0.00	0.00
#1 Causticizer	67.36	71.61	71.11	79.70	85.79	85.09
#3 Causticizer	69.02	73.43	73.50	80.40	86.54	87.07
White Liquor	69.61	74.08	74.14	80.94	87.12	87.24
Weak Wash	15.98	16.58	17.03	19.18	20.73	20.47
Green Liquor	23.57	24.54	24.21	32.73	35.34	35.56

SAMPLE	TTA*			%TDS		
	NIR	CC	LAB	NIR	CC	LAB
Water	0.00	0.00	0.00	0.00	0.00	0.00
#1 Causticizer	108.52	103.88	103.75	20.48	17.95	17.98
#3 Causticizer	107.98	103.30	102.40	19.89	17.44	17.24
White Liquor	106.69	101.93	101.91	19.51	17.10	16.94
Weak Wash	31.83	28.03	27.88	N/A	N/A	N/A
Green Liquor	101.01	95.93	96.12	22.94	20.11	20.39

SAMPLE	$NaOH^*$			Na_2S^*		
	NIR	CC	LAB	NIR	CC	LAB
Water	0.00	0.00	0.00	0.00	0.00	0.00
#1 Causticizer	55.02	57.43	57.13	24.68	28.36	27.96
#3 Causticizer	57.64	60.32	59.93	22.76	26.22	27.14
White Liquor	58.28	61.04	61.04	22.66	26.08	26.20
Weak Wash	12.78	12.43	13.59	6.40	8.30	6.88
Green Liquor	14.41	13.74	12.86	18.32	21.60	22.70

SAMPLE	$Na_2CO_3^*$			%Sufidity (AA-basis)		
	NIR	CC	LAB	NIR	CC	LAB
Water	0.00	0.00	0.00	—	—	—
#1 Causticizer	28.82	18.09	18.66	30.97	33.06	32.86
#3 Causticizer	27.58	16.76	15.33	28.31	30.30	31.17
White Liquor	25.75	14.81	14.67	28.00	29.93	30.03
Weak Wash	12.65	7.30	7.41	33.37	40.04	33.61
Green Liquor	68.28	60.59	60.56	55.97	61.11	63.84

SAMPLE	%Sufidity (TTA-basis)			%CE		
	NIR	CC	LAB	NIR	CC	LAB
Water	—	—	—	—	—	—
#1 Causticizer	22.74	27.30	26.95	65.63	76.05	75.38
#3 Causticizer	21.08	25.38	26.50	67.64	78.25	79.63
White Liquor	21.24	25.58	25.71	69.36	80.47	80.62
Weak Wash	20.11	29.61	24.68	50.26	63.00	64.71
Green Liquor	18.14	22.51	23.62	17.43	18.49	17.52

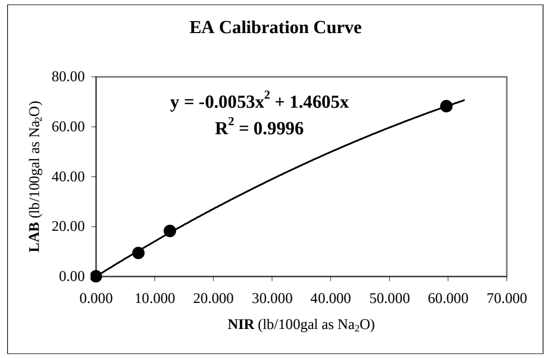
*Units in lb/100gal as Na_2O

Table A.6-1 compares the analyzer values before and after curve correction with the measured lab values. The water zeroed analyzer values are listed under "NIR", calibration curve corrected values are listed under "CC", and the laboratory measured values are listed under "LAB". This illustrates the effectiveness of the calibration curve in its ability to tune the base calibration models to lab measurements.

<u>Example A.6-2</u>:

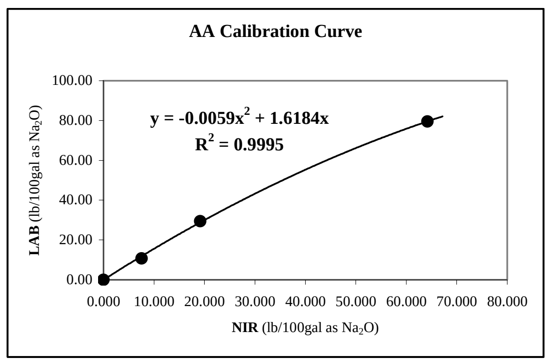
	g/L as Na_2O	lb/100gal as Na_2O	
SAMPLE	**EA_LAB**	**EA_NIR**	**EA_LAB**
Water	0.00	0.000	0.00
Weak Wash	11.22	7.216	9.36
Green Liquor	21.77	12.631	18.17
White Liquor	81.70	59.733	68.18

$$x_{cc,EA} = -0.0053\,x_{nz,EA}^2 + 1.4605\,x_{nz,EA}$$



	g/L as Na_2O	lb/100gal as Na_2O	
SAMPLE	**AA_LAB**	**AA_NIR**	**AA_LAB**
Water	0.00	0.000	0.00
Weak Wash	12.81	7.534	10.69
Green Liquor	35.24	19.152	29.41
White Liquor	95.30	64.179	79.53

$$x_{cc,AA} = -0.0059\,x_{nz,AA}^2 + 1.6184\,x_{nz,AA}$$



	g/L as Na_2O	lb/100gal as Na_2O	
SAMPLE	**TTA_LAB**	**TTA_NIR**	**TTA_LAB**
Water	0.00	0.000	0.00
Weak Wash	14.42	11.718	12.03
Green Liquor	114.05	89.713	95.18
White Liquor	110.95	85.918	92.59

$$x_{cc,TTA} = 0.0002\,x_{nz,TTA}^2 + 1.0485\,x_{nz,TTA}$$

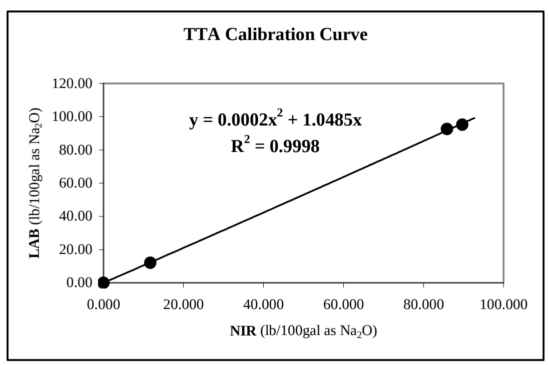


Figure A.6-3. Calibration curves for Example A.6-2.

Figure A.6-3 depicts calibration curves for weak wash, green liquor, and white liquor where the lab values are initially reported in g/L as Na_2O. The procedure is analogous to

the previous example except the lab values are converted from g/L as Na_2O to lb/100gal as Na_2O. XY scatter plots are generated for EA, AA, and TTA where the "NIR" values and "LAB" values in lb/100gal as Na_2O are used for the "X" and "Y" values respectively.

Final Notes:

- Since laboratory measurements can have a significant amount of variability (in terms of repeatability), better calibration curves will be achieved by utilizing more calibration samples.
- Make sure the Normal Operating Procedure is followed when analyzing samples.
- Sometimes a linear fit is better than a 2nd order polynomial.
- In the absence of values that fall between the minimum and maximum, it is best to use a linear fit.
- Contact R. E. Hodges, LLC for any further assistance.

Appendix A.7 Automated Calibration Curve Determination

<u>Background</u>:

The ***DURALYZER-NIR™*** laboratory liquor analyzer has seven standard calibration (predictive) models for analyzing green, white, and black liquor samples. The calibration models have been developed through a combination of signal processing steps and mathematical regression techniques. Models are tuned by utilizing a calibration curve. Employing a calibration curve allows the individual component (EA, AA, etc.) models to be adjusted to match the lab test method (results) in a least squares sense. The seven models are as follows:

1. EA (REA for black liquor)
2. AA (RAA for black liquor)
3. TTA
4. Na_2SO_4
5. % TDS
6. % TDD
7. % TDO

Certain measurements may not be required for a given application of the analyzer. In which case, determining a calibration curve for that model would be unnecessary. For example, if there is not an interest in Reduction Efficiency then there is not a need for a sodium sulfate calibration curve. Likewise, %TDO (total dissolved organics) would only have meaning for black liquor samples.

For *automated* determination, each calibration curve will be represented by the second order polynomial given by Equation A.7-1.

$$x_{cc} = Ax_{ra}^2 + Bx_{ra} + C \qquad \text{(A.7-1)}$$

Constant curve parameters, *A* and *B*, are determined by the automated calibration curve procedure, x_{cc} is the "curve corrected" measurement value, and x_{ra} is the "raw" analyzer value prior to water zeroing, curve correction and bias adjustment. Both x_{cc} and x_{ra} must be in lb/100gal as Na_2O. The base units for the EA, AA, TTA, and Na_2SO_4 models are lb/100gal. Water sets the zero value for each curve, which is the *C* parameter in Equation A.7-1. Each calibration model will have its own set of *A*, *B* and *C* curve parameters.

<u>Samples</u>:

Follow the *Normal Operating Procedure* (Appendix A.5) relating to preparing and drawing samples into the cuvette. At a minimum, two liquor samples and one water sample (3 total samples) are required for calibration curve parameter determination. If, the current lab testing method in place produces less than desirable repeatability, more than two liquor samples will be required. It is important to note that all samples need to be tested using the same method and by the same person. This eliminates potential of conflicting biases being introduced into the lab values and ultimately will yield a less

than optimal calibration curve. Special care should be taken when performing the lab tests to ensure the highest possible precision in the measurement values.

For example, past experience has shown that using an autotitrator for the white liquor "ABC" test and manual titration for the green liquor "ABC" test is not a good idea. If this is the standard treatment at a mill, run the green and white liquor tests for curve parameter determination on the autotitrator. After the calibration curves are set and in use, go back and run manual tests on the green liquor and make any adjustments by using the "Set Bias" function on the LCD panel *Main Menu*. Details on setting biases can be found in Section 4.4.5 and Appendix A.8.

For EA and AA, a white liquor and a green liquor sample is recommended to determine the curve parameters. A weak wash sample and/or a black liquor sample can be used as well. The important idea is to have samples that give values across a wide range and between the upper and lower values (i.e., green liquor EA and AA falls between water and white liquor). Black liquor with very low REA or high organic and soap content is not ideal for curve parameter determination. In this case, any black liquor measurement adjustments should be made through the "Set Bias" function. If there is only a white liquor sample available, then a white liquor sample may be diluted to form a second liquor sample. A one part white liquor to one part water dilution ratio would work fine and yield an EA and AA value roughly half of the undiluted sample. The dilute sample must still be tested to obtain a precise lab measurement value (do not assume it is exactly half the undiluted value).

The TTA calibration curve can be determined from green liquor, weak wash, and white liquor samples. Other combinations are possible as well, such as weak wash and white liquor or white liquor and diluted white liquor (see above paragraph for comments on dilution).

Reduction efficiency (RE%) computation requires the measurement of sodium sulfate. Green liquor samples are used to determine the Na_2SO_4 curve parameters. Obtain a single green liquor sample in an amount suitable to divide into three samples of equal volume. Add approximately 5 g/L and 10 g/L of $Na_2SO_{4(s)}$ to samples one and two respectively. Do not add any to the third sample. Allow the sodium sulfate added to samples one and two to completely dissolve (this will take more time the cooler the sample is). Next, test all three samples using the standard laboratory test procedure for sodium sulfate in green liquor.

Determination of the %TDS calibration curve parameters can be carried out using white, green and black liquors. A convenient relationship for calculating %TDS in white and green liquor is depicted in Equation A.7-2.

$$\%TDS = (\rho - 1.000) \cdot 100\% \tag{A.7-2}$$

The density (ρ) is in g/cm^3 and can be measured by a handheld density meter or by a gravimetric method. At room temperature, measure out a precise volume of sample (V)

in mL then determine the mass (*m*) in grams. The black liquor %TDS is usually measured by a moisture analyzer or by oven drying.

Only green and white liquor samples are used to calculate the %TDD calibration curve. Lab values for %TDD can be calculated from the following equation if the EA, AA, TTA, ρ, and %TDS values are known.

$$\%TDD = \%TDS - \left[\frac{(1.291 * NaOH) + (1.259 * Na_2S) + (1.710 * Na_2CO_3)}{\rho * 1000 * 0.834} \right] \qquad \text{(A.7-3)}$$

Density (ρ) is in g/cm^3, whereas NaOH, Na_2S, and Na_2CO_3 are in lb/100gal as Na_2O.

Determining the %TDO calibration curve requires black liquor samples with a known %TDS. A precisely measured mass of black liquor is dried and burned in a furnace to measure the total dissolved organics. The %TDO is computed by Equation A.7-4.

$$\%TDO = \%TDS - \left(\frac{remaining\ mass\ of\ burned\ sample}{mass\ of\ wet\ sample} \right) \cdot 100 \qquad \text{(A.7-4)}$$

<u>Procedure</u>:

1. Make sure the analyzer has been **powered on for a minimum of 2 hours (12+ hours recommended)** to allow for the analyzer to reach thermal equilibrium and stabilize.
2. Collect all liquor samples that will be required for determining the calibration curve parameters.
3. Perform the necessary laboratory tests on the collected samples. The tests will yield the "LAB" values.
4. After lab testing is complete, access the *Main Menu* and select "Calibrate" to open the *Calibrate Curve, Slope and Offset* screen. Once the calibration procedure has been started, **do not** exit the *Calibrate Curve, Slope and Offset* screen until the procedure is completed. Otherwise, sample spectral data and component values will be lost.
5. Test the collected samples on the ***DURALYZER-NIR™*** laboratory liquor analyzer following basically the same procedures set forth in Section 4.3 and Appendix A.5. Start with the water sample (make sure "Water" is selected on the top section of the screen). With water in the sample cuvette, press the "Scan" button. The current "Curve", "Slope", and "Offset" values for each component curve will be displayed. For the water sample, zeros are preloaded for each component. **Do not change these values.**
6. Eject the water sample and move to the next calibration sample by selecting "Sample 1" at the top of the screen.
7. Draw the sample into the cuvette (as outlined in the *Normal Operating Procedure*). Press the "Scan" button to analyze the sample. As with the water, the analyzer values will be displayed under the "Scan" button. **Do not** be

concerned if the analyzer values look odd (possibly negative numbers). The calibration procedure will take care of positioning the calibration curve and adjusting its shape.

8. Enter the available lab measured values for sample 1 by pressing the corresponding button to the immediate right of the component. If a value is not available for a particular component, "NA" must be entered. **Entered concentration values must be in units of lb/100gal as Na_2O**. These are the base units for the EA, AA, TTA, and Na_2SO_4 calibration models. Conversion factors are provided on the "Help" screen for some commonly used units.
9. Repeat steps 6 – 8 until all of the calibration samples have been processed.
10. Entered lab test values can be reviewed for any sample by by pressing the corresponding button. Samples can also be rescanned if needed by selecting the corresponding sample button (at the top of the screen) , drawing the sample into the cuvette, and then pressing "Scan".
11. After all collected samples have been processed, select "Calibrate" at the bottom of the screen. New "Curve" "Slope" and "Offset" values will be calculated and displayed along with the correlation coefficient. Additionally, a hard copy summarizing the calibration data and results will be printed out (provided the printer is connected and powered on).
12. Review the results. If necessary, make changes such as rescanning a sample or changing entered values. Press the "Calibrate" button again to generate updated calibration curves if any changes were made.
13. Once the calibration curve procedure is complete, press the "Back" button to return to the *Main Menu* screen. The calibration curve parameters can be accessed and viewed by selecting "Slope/Offset" from the *Main Menu*.

Figure A.7-1 illustrates the menu navigation for automated calibration curve determination. Starting from the *Operator Screen* (1), "About" is selected and screen (2) appears. Pressing "TM" brings up screen (3) to enter the password which allows access to the *Main Menu*, screen (4). From the *Main Menu* (4), "Calibrate" is selected and the calibration parameters are calculated using the *Calibrate Curve, Slope and Offset* screen (5). The current sample is selected at (A) and then scanned (B). After the NIR spectrum has been acquired, the lab measured values are entered (C). Once all of the collected samples have been processed, "Calibrate" is pressed (D).

Understanding of the inner workings of the automated calibration curve determination can be reinforced by examining the examples in Appendix A.6 Manual Calibration Curve Determination. This basically illustrates the calculations that occur "behind the scenes" in the automated procedure. The examples also highlight the improvements made after calibration curves are implemented.

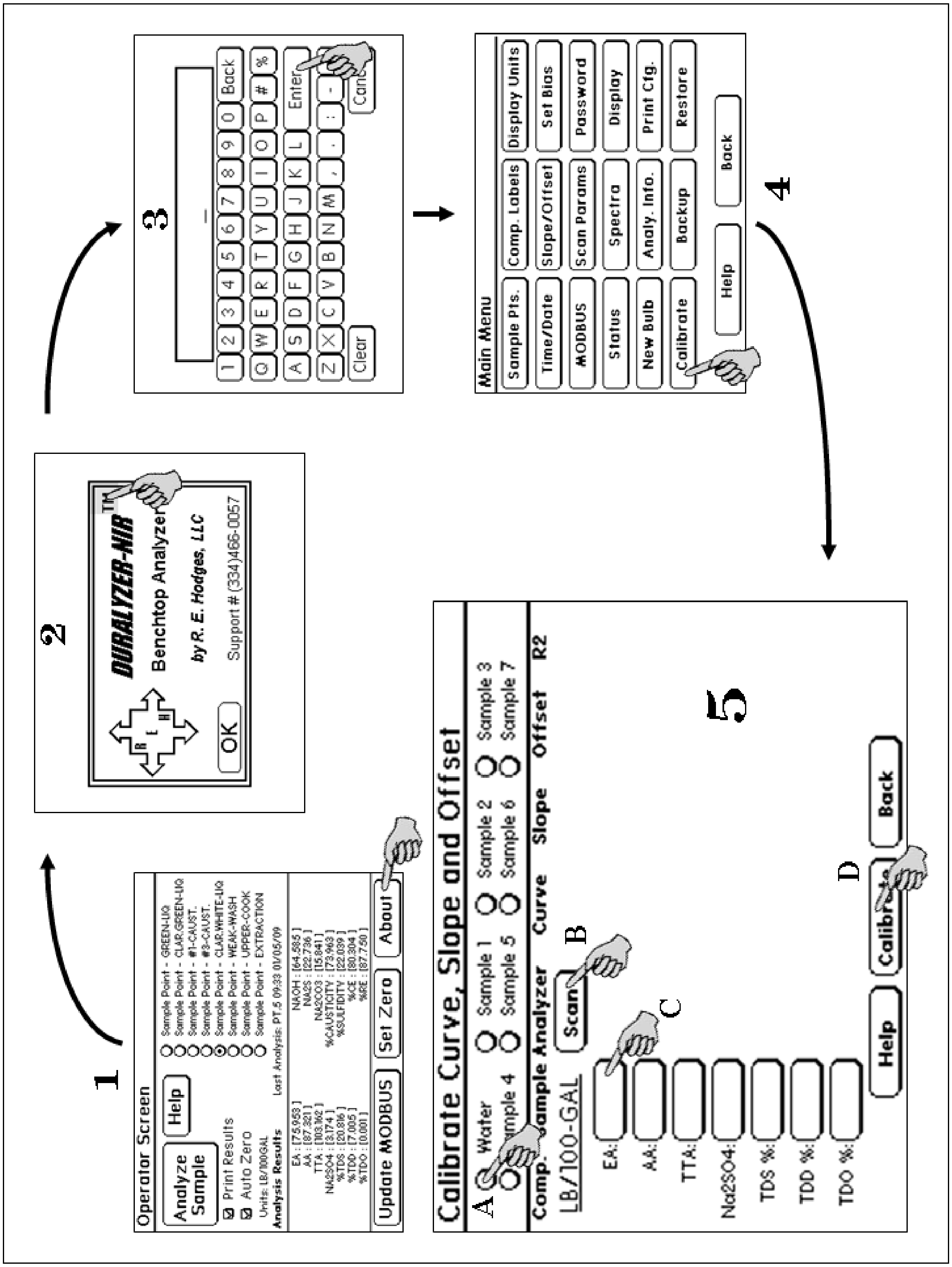


Figure A.7-1. Menu sequence for automated calibration curve determination.

Final Notes:

- Since laboratory measurements can have a significant amount of variability (in terms of repeatability), better calibration curves will be achieved by utilizing more calibration samples.
- Make sure the Normal Operating Procedure for preparing and drawing samples into the cuvette.
- In the absence of values for a particular component, remember to enter “NA”.
- Once the calibration procedure has been started, **do not** exit the *Calibrate Curve, Slope and Offset* screen until the procedure is completed.
- Entered concentration values must be in units of **lb/100gal as Na_2O**. These are the base units for the EA, AA, TTA, and Na_2SO_4 calibration models.
- Contact R. E. Hodges, LLC for any further assistance.

Appendix A.8 Bias Adjustment

Background:

Another method for tuning of the ***DURALYZER-NIR™*** laboratory liquor analyzer measurements is implementing bias adjustments. Bias adjustments can be used in conjunction with calibration curves or as a standalone tuning method. Every measured component for each sample point can have a scale and/or offset correction to bias the analyzer in order to match the manual lab tests performed onsite. If calibration curves have been generated and are in use, the bias corrections will usually be minimal or nonexistent. Bias adjustment is represented by the following equation

$$x_b = S_1 x_{cc} + S_0 \qquad \text{(A.8-1)}$$

where x_b is the bias corrected (adjusted) value, x_{cc} is the calibration curve corrected value, S_1 is the bias slope, and S_0 is the bias offset (S_1 and S_0 are the bias adjustment parameters). Both x_b and x_{cc} are in the current display units (unlike calibration curves, which must be determined based on lb/100gal). Bias adjustments can be applied in three ways: (1) slope and offset, (2) slope only (S_0 = 0), and (3) offset only (S_1 = 0). The data will dictate the best choice. Two examples are presented to highlight the bias adjustment procedure.

Samples:

Follow the *Normal Operating Procedure* (Appendix A.5) for analyzing the samples. At a minimum, three liquor samples are required to determine the bias adjustment for a particular sample point. Water is **not** used when determining bias adjustments. If, the current lab testing method in place produces less than desirable repeatability, more than three liquor samples will be required. It is important to note that all samples need to be tested using the same method and by the same person. This eliminates potential of conflicting biases being introduced into the lab values. Special care should be taken when performing the lab tests to ensure the highest possible precision in the measurement values.

Procedure:

1. Make sure the analyzer has been **powered on for a minimum of 2 hours (12+ hours recommended)** to allow for the analyzer to reach thermal equilibrium and stabilize. If calibration curves are to be used, perform that procedure before attempting any bias adjustment. Set the desired analyzer display units and make sure the current bias values are set to the default values (slope = 1 and offset = 0). See Sections 4.4.3 and 4.4.4 respectively.
2. Collect all liquor samples that will be required for determining the bias adjustment for a particular sample point.
3. Perform the necessary laboratory tests on the collected samples. The tests will yield the “LAB” values.

4. After lab testing is complete, set the water zero if this has not been performed recently. Refer to Section 4.3 *Normal Operation* and Appendix A.5 *Normal Operating Procedure*.
5. Test the collected samples on the ***DURALYZER-NIR™*** laboratory liquor analyzer following the procedures set forth in Section 4.3 and Appendix A.5. After each sample has been analyzed, record the values shown in the "Analysis Results" section of the *Operator Screen*. These will be the analyzer or "NIR" values. It is highly recommended to have the "Print Results" box checked on the *Operator Screen* and the printer connected prior to analyzing the samples.
6. After all collected samples have been lab tested and analyzer tested, the mathematical determination of the bias adjustment parameters is performed. This requires the use of spreadsheet software such as Microsoft® Excel.
7. On the spreadsheet, make three column headings for the sample, the analyzer measurement value and the lab measurement value.
8. Construct a scatter plot with the analyzer values being the "X" data and the lab values being the "Y" data. Fit the data with a linear trendline. The resulting trendline equation is the bias adjustment in the form of Equation A.8-1. In some cases, it may be preferable to bias adjust the data with just a slope correction (S_0 in Equation A.8-1 will be zero) or just an offset correction (S_1 in Equation A.8-1 will be zero).
9. Select "Set Bias" on the *Main Menu*. From the *Bias Adjustments* screen select the desired sample point to bias and press "Edit". At the *Edit Bias Values* screen, enter the appropriate "Slope" and "Offset" biases and press "Update" when finished. The component (value to be measured) is listed in the left column with the corresponding slope and offset biases to the right. Once "Update" has been pressed, the effects are immediate (meaning the display results on the LCD panel will be updated to reflect the new bias adjustment).
10. Repeat steps 2 – 9 for all sample points requiring a bias adjustment.

Two examples are presented to further highlight and explain the procedure and concepts presented above.

Example A.8-1:

In the following example, the black REA analyzer values need to be slightly tuned (biased) to agree with the laboratory measurements. A calibration curve is already in place and was determined based on white, green, and black liquor. The white and green liquor analyzer values are in agreement with the lab. However, low REA black liquor samples (containing an appreciable amount of soap) are slightly off from the lab and need fine tuning. Figure A.8-1 illustrates the determination of bias adjustment parameters for this case. Three black liquor samples (evaporator, chemiwasher, and secondary evaporator) are on the same analyzer sample point. Thus, these samples are used to determine the bias adjustment for REA. All values are in g/L as Na_2O, with the analyzer values under the heading "REA_NIR", lab values under "REA_LAB", and the bias corrected values under "REA_B". The resulting bias adjustment equation is displayed

with the data. Bias adjustment parameters are entered into the Edit Bias Values screen (Figure A.8-2).

	g/L as Na_2O		
SAMPLE	**REA_NIR**	**REA_LAB**	**REA_B**
Evaporator	3.14	4.03	4.04
Chemiwasher	2.34	3.04	3.07
Secondary Evaporator	2.58	3.41	3.37

$$x_{b,REA} = 1.2154\, x_{cc,REA} + 0.2271$$

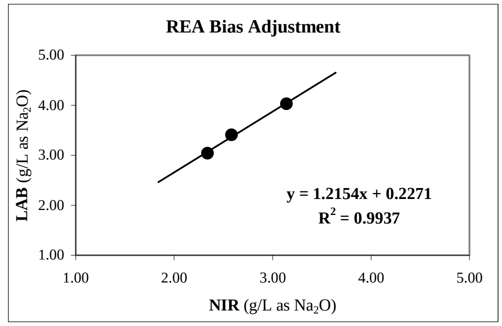


Figure A.8-1. Bias adjustment determination for Example A.8-1.

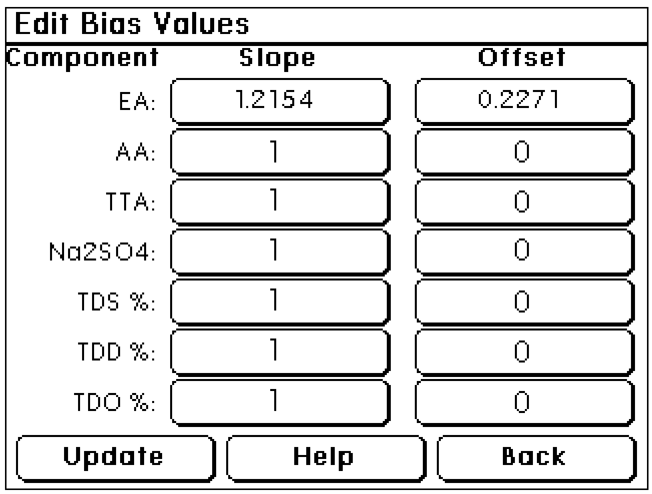


Figure A.8-2. Bias adjustment parameters for Example A.8-1.

Example A.8-2:

After implementing a TTA calibration curve based on green liquor, weak wash, clarified white liquor, and causticizer samples, the clarified white liquor analyzer measurements were slightly high compared to the lab. The TTA analyzer measurements for the other sample points were in agreement with the lab. Thus a bias adjustment was implemented on the clarified white liquor TTA measurement. Six total clarified white liquor samples were collected and tested over a 3 to 4 day period. The bias adjustment determination for this data set is illustrated in Figure A.8-3.

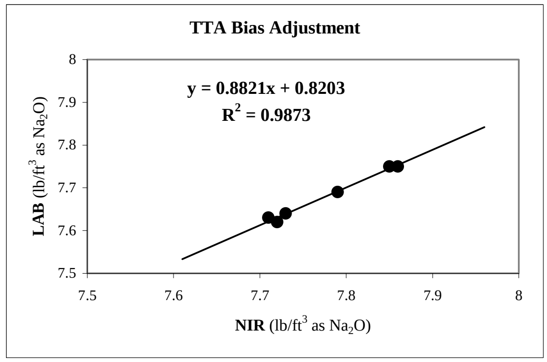
The three bias adjustment methods (1) slope and offset, (2) slope only (S_0 = 0), and (3) offset only (S_1 = 0) are considered. Table A.8-1 compares analyzer values before bias adjustment with the three bias adjustment methods and the lab values (TTA_NIR, TTA (1), TTA (2), TTA (3), and TTA_LAB respectively). Examination of Table A.8-1 shows all three bias corrections are in agreement with the lab measured values. In this instance,

all three would be an acceptable choice. Other cases may point favorably to a particular method.

SAMPLE	TTA_NIR (lb/ft^3 as Na_2O)	TTA_LAB (lb/ft^3 as Na_2O)
White Liquor 1	7.71	7.63
White Liquor 2	7.72	7.62
White Liquor 3	7.79	7.69
White Liquor 4	7.86	7.75
White Liquor 5	7.73	7.64
White Liquor 6	7.85	7.75

$$\boxed{x_{b,TTA} = 0.8821 x_{cc,TTA} + 0.8203}$$

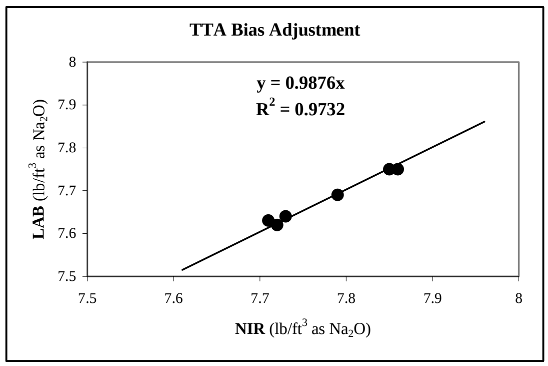
(1) Slope and Offset



SAMPLE	TTA_NIR (lb/ft^3 as Na_2O)	TTA_LAB (lb/ft^3 as Na_2O)
White Liquor 1	7.71	7.63
White Liquor 2	7.72	7.62
White Liquor 3	7.79	7.69
White Liquor 4	7.86	7.75
White Liquor 5	7.73	7.64
White Liquor 6	7.85	7.75

$$\boxed{x_{b,TTA} = 0.9876 x_{cc,TTA}}$$

(2) Slope Only



SAMPLE	TTA_NIR (lb/ft^3 as Na_2O)	TTA_LAB (lb/ft^3 as Na_2O)	ΔTTA (LAB-NIR) (lb/ft^3 as Na_2O)
White Liquor 1	7.71	7.63	-0.08
White Liquor 2	7.72	7.62	-0.10
White Liquor 3	7.79	7.69	-0.10
White Liquor 4	7.86	7.75	-0.11
White Liquor 5	7.73	7.64	-0.09
White Liquor 6	7.85	7.75	-0.10

Avg. Δ **-0.0967**

$$\boxed{x_{b,TTA} = x_{cc,TTA} - 0.0967}$$

(3) Offset Only

Figure A.8-3. Bias adjustment determination for Example A.8-2.

Table A.8-1. Bias adjustment method comparison for Example A.8-2.

SAMPLE	lb/ft^3 as Na_2O				
	TTA_NIR	TTA (1)	TTA (2)	TTA (3)	TTA_LAB
White Liquor 1	7.71	7.62	7.61	7.61	7.63
White Liquor 2	7.72	7.63	7.62	7.62	7.62
White Liquor 3	7.79	7.69	7.69	7.69	7.69
White Liquor 4	7.86	7.75	7.76	7.76	7.75
White Liquor 5	7.73	7.64	7.63	7.63	7.64
White Liquor 6	7.85	7.74	7.75	7.75	7.75

Figure A.8-4 illustrates the menu navigation for entering the bias adjustment parameters. Starting from the *Operator Screen* (1), “About” is selected and screen (2) appears. Pressing “TM” brings up screen (3) to enter the password which allows access to the *Main Menu* (4). From the *Main Menu* (4), “Set Bias” is selected and the sample point (to be biased) is chosen on the *Bias Adjustments* screen (5). The bias adjustment parameters are entered at point (A) on the *Edit Bias Values* screen (6). Once the bias parameters have been entered, press “Update” (B) for the changes to take effect.

Final Notes:

- Since laboratory measurements can have a significant amount of variability (in terms of repeatability), optimal bias adjustments will be achieved by utilizing more samples.
- Make sure the Normal Operating Procedure is followed when analyzing samples.
- Sometimes only a slope **or** offset correction is required.
- In the absence of values that fall over a wide range, it may be safer to employ only a slope **or** offset.
- In most cases, bias adjustment will not be needed if calibration curves are in place.
- Contact R. E. Hodges, LLC for any further assistance.

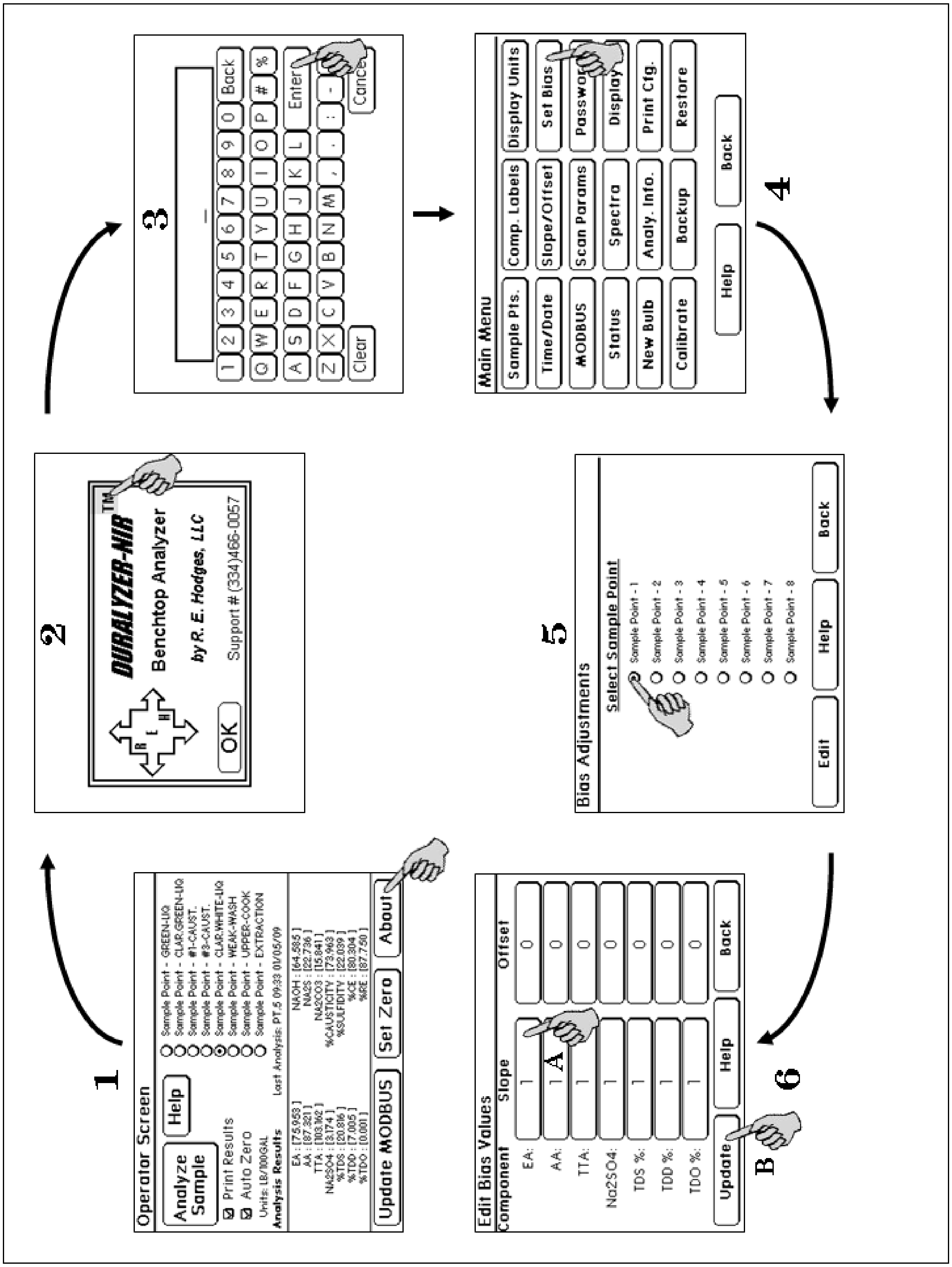


Figure A.8-4. Menu sequence for implementing bias adjustments.

Appendix A.9 Printer Details

The ***DURALYZER-NIR™*** laboratory liquor analyzer includes a Star Micronics TSP600 Series Thermal Printer. Connection to the analyzer is accomplished via the RS232 port. The printer is equipped with its own power unit and power cord which connects to any standard 3 prong outlet. Further details not described in this appendix can be found on the Star Micronics webpage.

Connecting:

The printer connections should be made in the following order before engaging the power switch (located on the lower left side): (1) connect to the analyzer via the RS232 port, (2) plug the DC end of the power cord into the printer, and (3) plug the 3 prong AC end into a standard wall outlet. Cables and other images of the printer are shown in Figure A.9-1.

Paper Roll Installation:

The thermal paper roll must have a width of 80 mm, thickness of 65 to 85 μm, and a roll diameter of 90 mm (or less).

1. Open the printer cover by pushing the lever on the top front of the printer (highlighted in Figure A.9-1).
2. Place the paper roll in and pull the leading edge of the paper towards the front of the printer (paper direction should be such that the leading edge is coming from under the roll, not over top).
3. Close the printer cover by pressing down on both sides.

Cleaning and Care:

The presence of accumulated dust will adversely affect printer performance, possibly causing the printout to be unreadable or not to function at all. Cleaning the printer is a three part process: (1) Cleaning the thermal head, (2) Cleaning the paper holder, and (3) Cleaning the paper housing. Care must be taken when cleaning the thermal head since it is easy to damage. Use isopropyl alcohol and a soft, clean cloth. A soft cloth is sufficient for removal of dust from the paper holder. The external housing of the printer can be wiped off with a moist cloth.

Specifications:

- Dimensions: 199 mm × 142 mm × 132 mm (see Figure A.9-1)
- Weight: 1.4 kg
- Paper width: 80 mm
- Maximum print speed: 100 mm/s (800 dots/s)
- Dot density: 8 dots/mm (203 dpi)
- Input Voltage: 24 Volts DC
- Power Unit Input Voltage: 100 to 240 Volts (50 to 60 Hz)

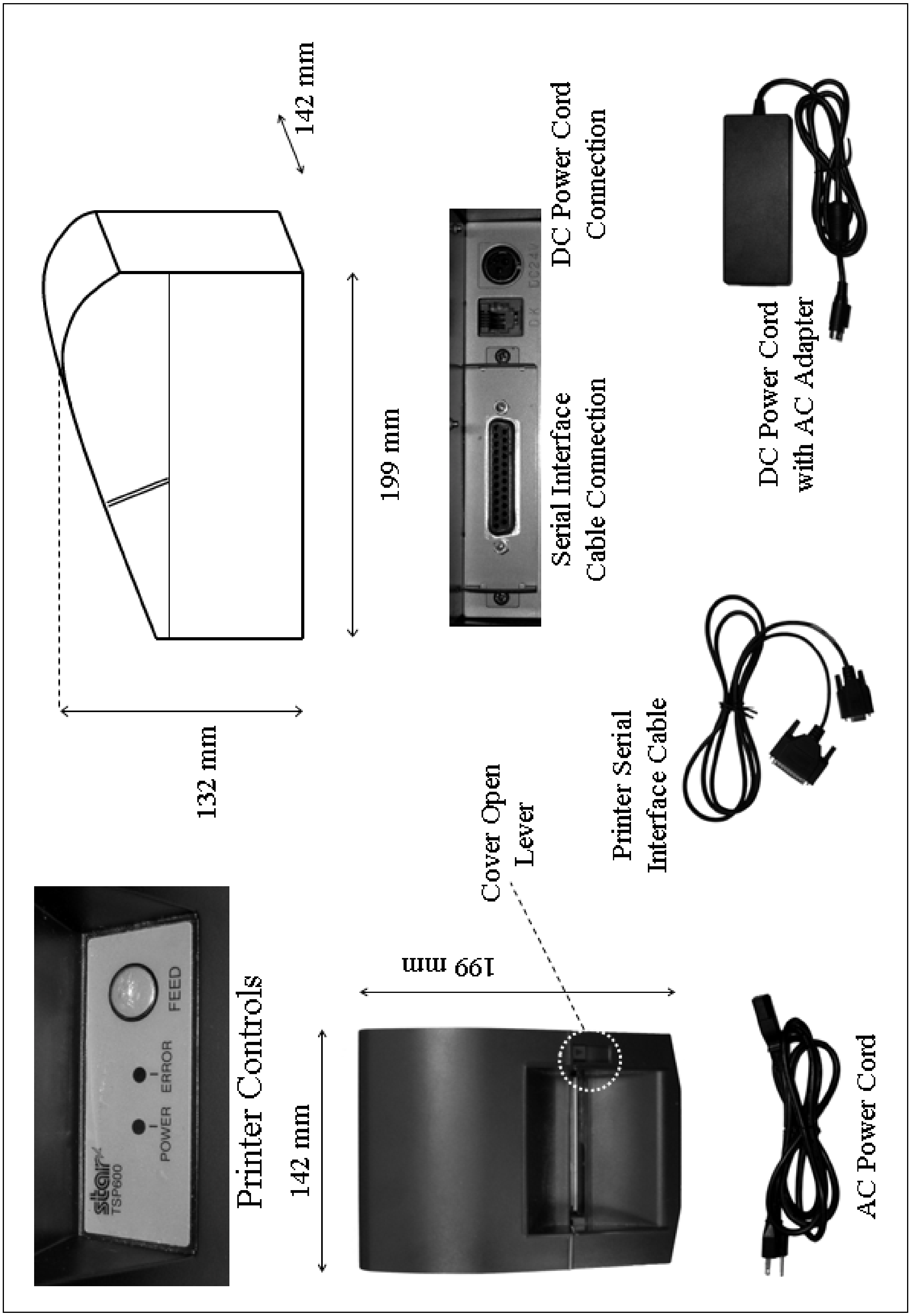


Figure A.9-1. Printer images.

Appendix A.10 NIR Light Source Replacement

The maintenance requirements for the ***DURALYZER-NIR™*** laboratory liquor analyzer are minimal both in terms of time and cost. All maintenance tasks should only be performed by qualified personnel that have read and understand this manual. The typical lifespan of the QTH NIR light source should be approximately one year. Before the bulb completely expires, there are usually signs the end of lifespan is approaching. A drop in bulb intensity combined with erratic measurements will be the most noteworthy characteristics. It is recommended that a replacement bulb be ordered and kept on hand.

<u>Replacement Steps</u>:

1. Power off the analyzer (Figure A.10-1)
2. Remove the light source enclosure cover (Figure A.10-2)
3. Loosen the light source holder set screw (Figure A.10-3)
4. Unplug the old bulb and remove
5. Plug in the new bulb
6. Insert the new bulb into the light source holder and tighten the set screw
7. Power on the analyzer and replace the enclosure cover
8. Allow the new bulb to stabilize for at least 2 hours
9. Utilize the "New Bulb" function from the Main Menu to test for detector saturation. Adjust the bulb position in the light source holder if necessary.

<u>Further Details</u>:

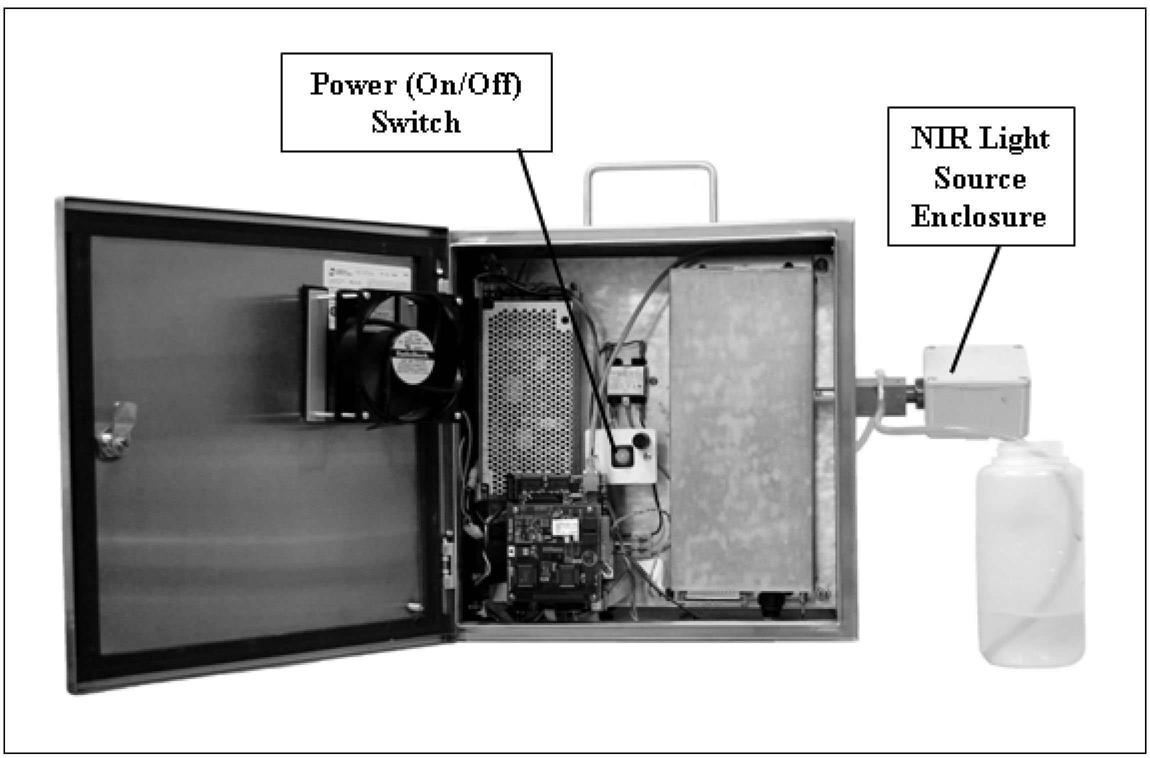


Figure A.10-1. Power switch and light source enclosure locations.

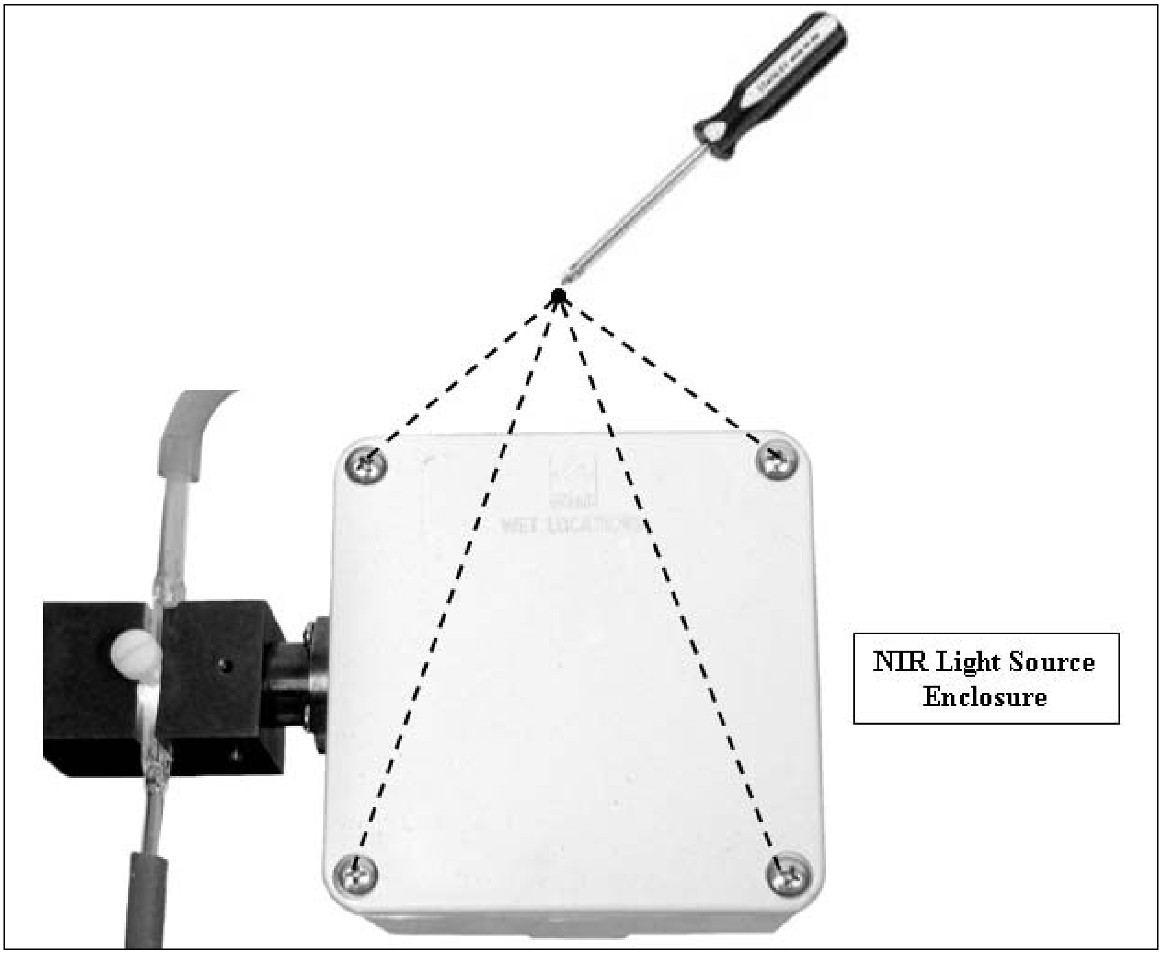


Figure A.10-2. Light source enclosure cover removal.

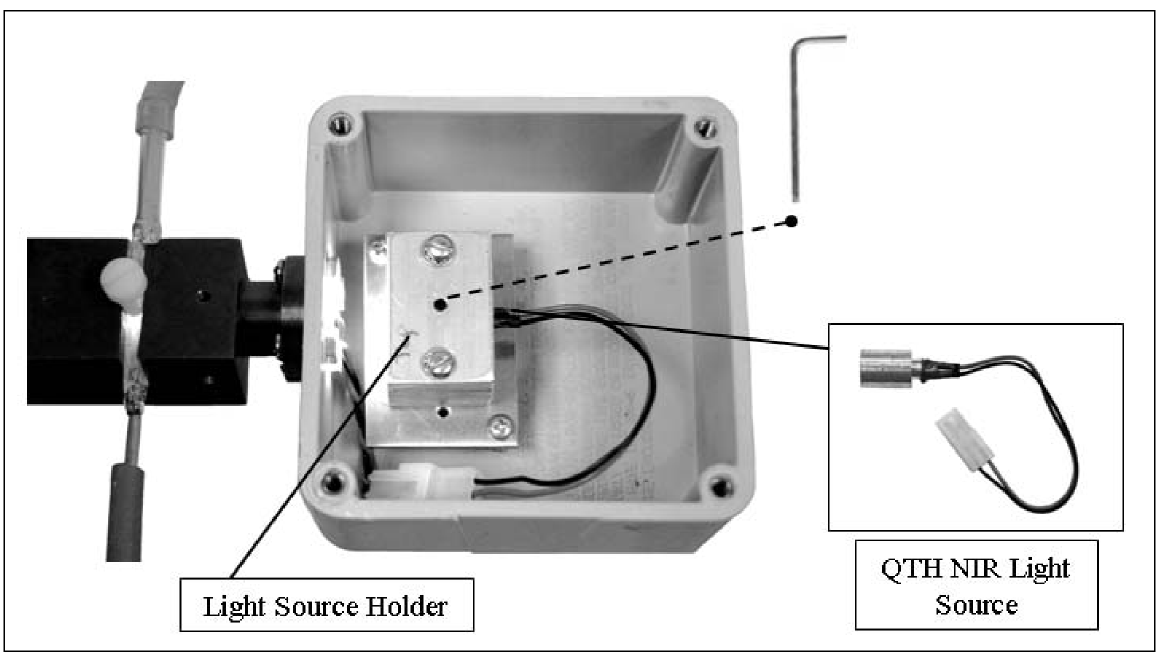


Figure A.10-3. NIR light source removal.

Replacement of the NIR light source (QTH light bulb with reflective coating) may require a slight adjustment after installation. The "New Bulb" function on the *Main Menu* will access the *New Bulb Adjustment* screen. After the bulb has been replaced, draw water into the sample cuvette. Select the "Trigger Scan" option on the left side of the screen to check if the new bulb saturates the detector. Figures A.10-4 and A.10-5 show a water scan without saturation and a water scan in which the detector is saturated. An unsaturated water scan (Figure A.10-4) indicates the replacement bulb is positioned and functioning correctly. If the water scan resembles the spectrum shown in Figure A.10-5 (top portion of the two peaks are "cut off" meaning transmission has reached 100%), the detector is saturated. Detector saturation occurs even if only one wavelength is 100%. The desired transmission % is between 75 and 85 for the highest spectral peak. The remedy for detector saturation is to slightly reposition the bulb (by rotation and/or relative location in the light source holder). Menu sequencing for the "New Bulb" function is shown in Figure A.10-6.

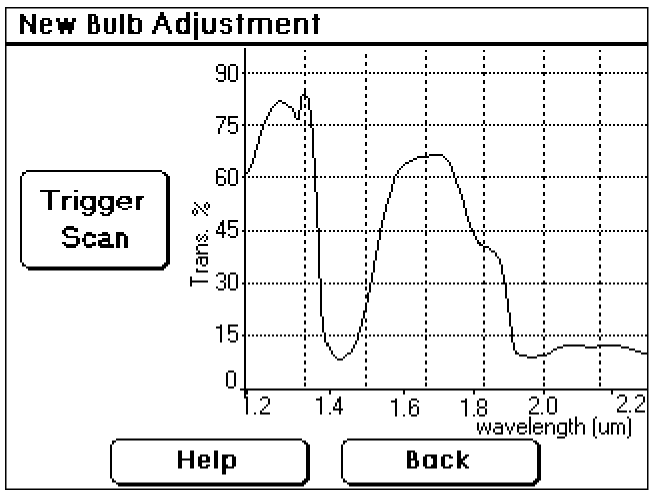


Figure A.10-4. Unsaturated water spectrum.

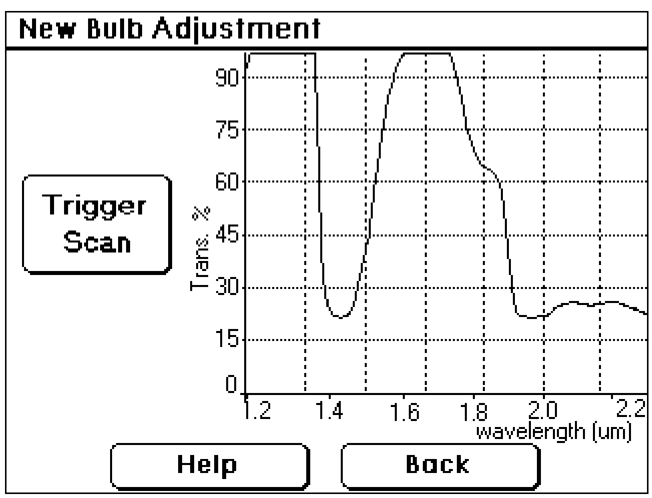


Figure A.10-5. Saturated water spectrum.

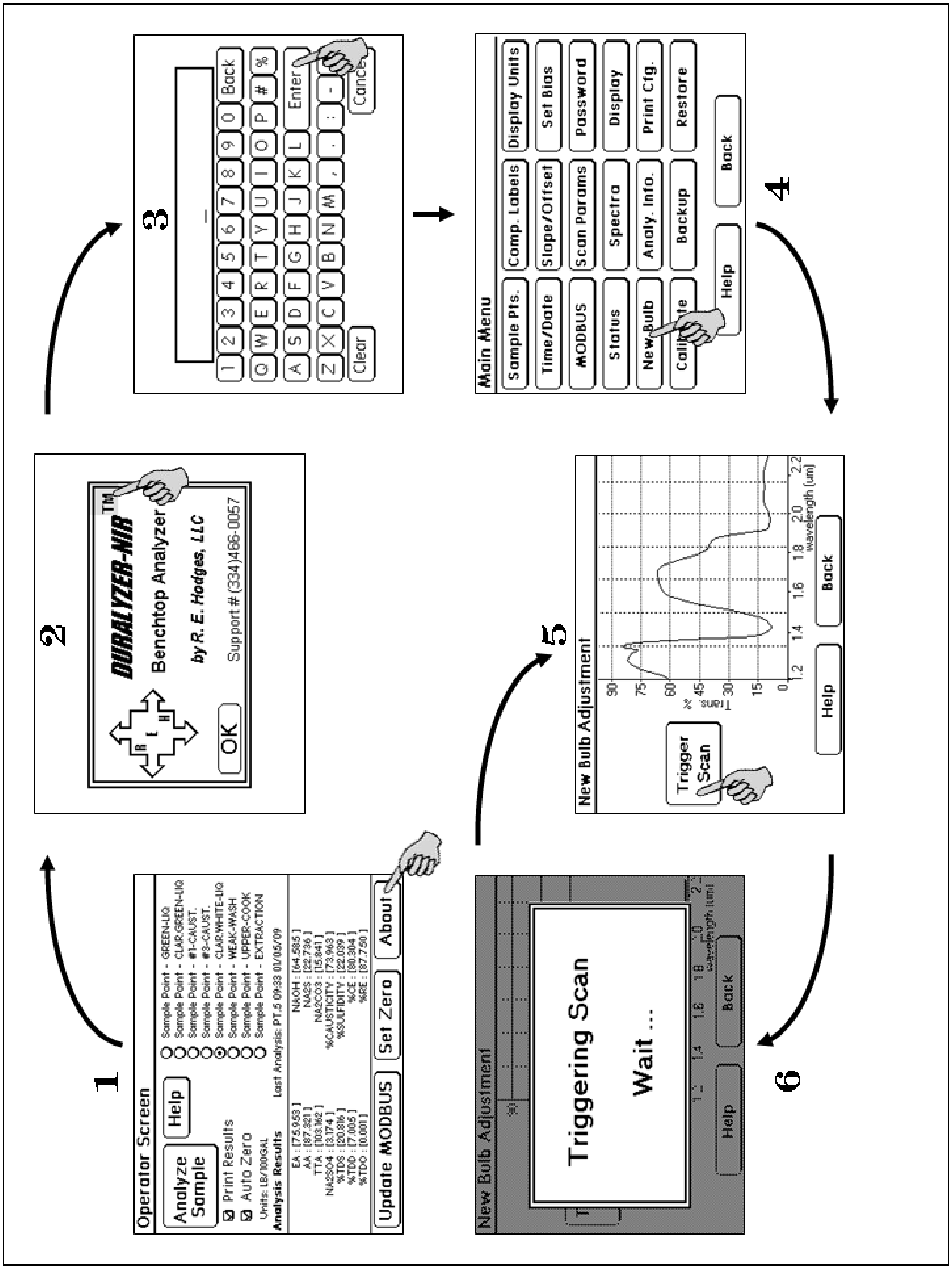


Figure A.10-6. Menu sequence for the “New Bulb” function.

Appendix A.11 MODBUS

The ***DURALYZER-NIR™*** laboratory liquor analyzer is equipped with MODBUS communications ability. It is configured by selecting "MODBUS" on the *Main Menu*. This will pull up the *MODBUS Configuration* screen, shown in Figure A.11-1 with example settings. The settings on this screen are used to configure the MODBUS TCP interface to the analyzer. Simply touch the box to be edited and enter the new value using the virtual LCD keyboard. Entries must conform to the format displayed above each box. Figure A.11-2 illustrates the LCD menu sequencing for configuring the MODBUS and Table A.11-1 lists the MODBUS register addresses.

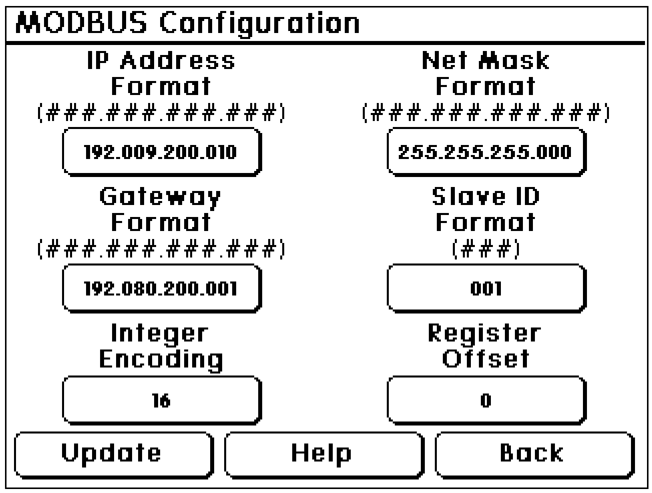


Figure A.11-1. *MODBUS Configuration* screen.

- "IP Address": refers to the IP address assigned to the analyzer (Class C address,32 bit)
- "Net Mask": mask for the IP address (255.255.255.000)
- "Gateway": IP address assigned to device with which analyzer is communicating (without the presence of a router, this will usually be 000.000.000.000)
- "Slave ID": the analyzer will always be the "slave" or "server" and can have an ID ranging from 001 to 247 (as defined by MODBUS specifications)
- "Integer Encoding": will be 16 unless the DCS interprets the higher order bit as a sign bit, in which case it will be 15
- "Register Offset": will be 0 if register 1 is address 0 for the MODBUS driver used by the DCS, otherwise this will be 1 or -1 (some devices refer to address instead of register number)

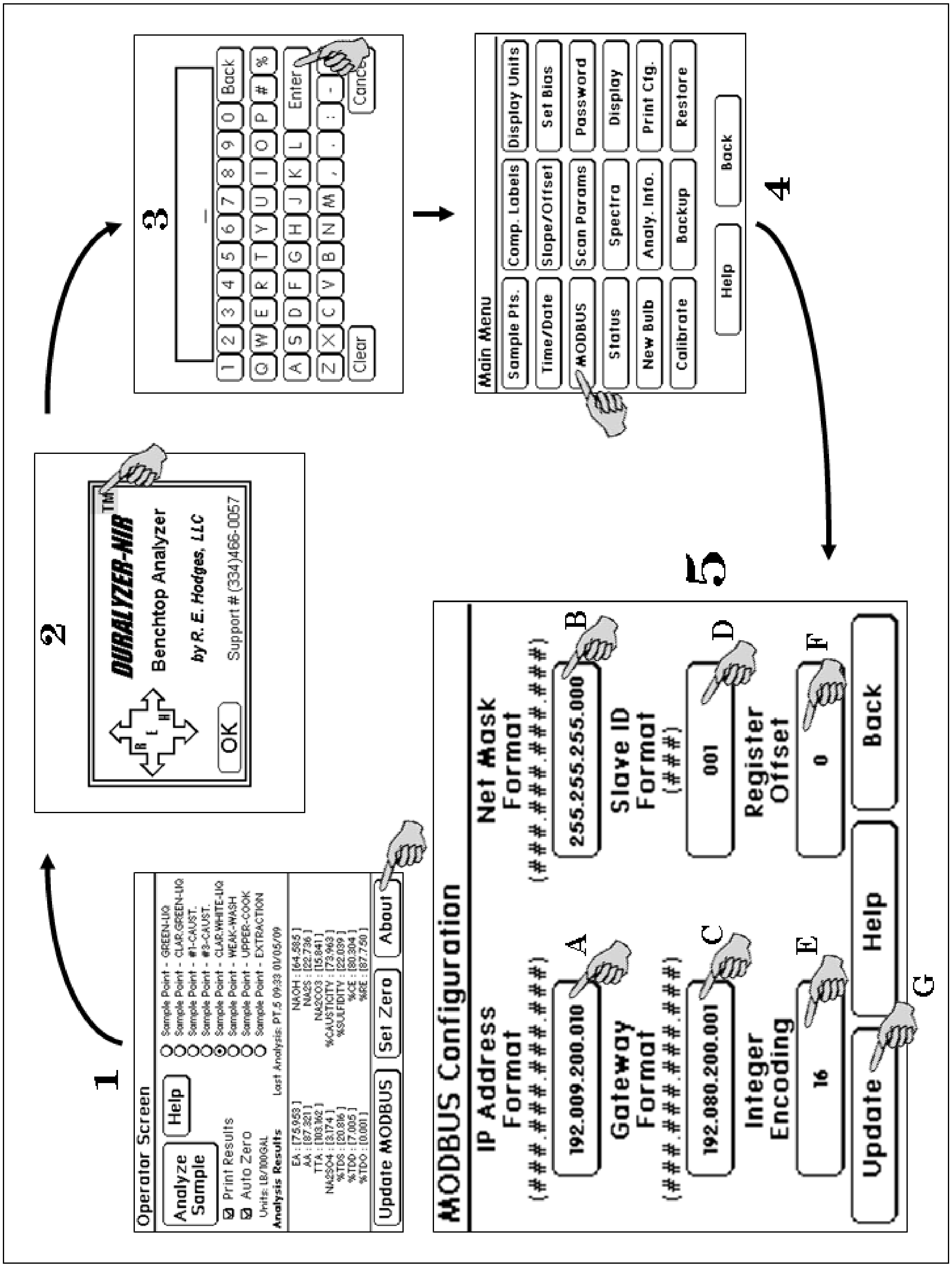


Figure A.11-2. Menu sequence for MODBUS configuration.

Figure A.11-2 illustrates the menu navigation for entering the MODBUS configuration settings. Starting from the *Operator Screen* (1), "About" is selected and screen (2) appears. Pressing "TM" brings up screen (3) to enter the password which allows access

to the *Main Menu* (4). From the *Main Menu* (4), “MODBUS” is selected and the MODBUS settings are entered on the *MODBUS Configuration* screen (5). Screen (5) is divided into steps A through G. Once the settings have been entered (A – F), select “Update” (G) for the changes to take effect.

Table A.11-1. MODBUS Register Addresses.

MODBUS Register Addresses ('4XXXX')								
Register	**Description**		**Register**	**Description**		**Register**	**Description**	
40000	EA/REA Word	**Sample Point 1**	40045	EA/REA Word	**Sample Point 4**	40090	EA/REA Word	**Sample Point 7**
40001	AA/RAA Word		40046	AA/RAA Word		40091	AA/RAA Word	
40002	TTA Word		40047	TTA Word		40092	TTA Word	
40003	TDS Word		40048	TDS Word		40093	TDS Word	
40004	TDD Word		40049	TDD Word		40094	TDD Word	
40005	TDO Word		40050	TDO Word		40095	TDO Word	
40006	Na_2SO_4 Word		40051	Na_2SO_4 Word		40096	Na_2SO_4 Word	
40007	Na_2SO_3 Word		40052	Na_2SO_3 Word		40097	Na_2SO_3 Word	
40008	Na_2S_2O3 Word		40053	Na_2S_2O3 Word		40098	$Na_2S_2O_3$ Word	
40009	Cl^- Word		40054	Cl^- Word		40099	Cl^- Word	
40010	Sample Temperature Word		40055	Sample Temperature Word		40100	Sample Temperature Word	
40011	Reserved Word		40056	Reserved Word		40101	Reserved Word	
40012	Reserved Word		40057	Reserved Word		40102	Reserved Word	
40013	Reserved Word		40058	Reserved Word		40103	Reserved Word	
40014	Reserved Word		40059	Reserved Word		40104	Reserved Word	
40015	EA/REA Word	**Sample Point 2**	40060	EA/REA Word	**Sample Point 5**	40105	EA/REA Word	**Sample Point 8**
40016	AA/RAA Word		40061	AA/RAA Word		40106	AA/RAA Word	
40017	TTA Word		40062	TTA Word		40107	TTA Word	
40018	TDS Word		40063	TDS Word		40108	TDS Word	
40019	TDD Word		40064	TDD Word		40109	TDD Word	
40020	TDO Word		40065	TDO Word		40110	TDO Word	
40021	Na_2SO_4 Word		40066	Na_2SO_4 Word		40111	Na_2SO_4 Word	
40022	Na_2SO_3 Word		40067	Na_2SO_3 Word		40112	Na_2SO_3 Word	
40023	Na_2S_2O3 Word		40068	$Na_2S_2O_3$ Word		40113	$Na_2S_2O_3$ Word	
40024	Cl^- Word		40069	Cl^- Word		40114	Cl^- Word	
40025	Sample Temperature Word		40070	Sample Temperature Word		40115	Sample Temperature Word	
40026	Reserved Word		40071	Reserved Word		40116	Reserved Word	
40027	Reserved Word		40072	Reserved Word		40117	Reserved Word	
40028	Reserved Word		40073	Reserved Word		40118	Reserved Word	
40029	Reserved Word		40074	Reserved Word		40119	Reserved Word	
40030	EA/REA Word	**Sample Point 3**	40075	EA/REA Word	**Sample Point 6**	40120	Alarm Bit Field (Bits 15-0)	
40031	AA/RAA Word		40076	AA/RAA Word		40121	Alarm Bit Field (Bits 31-16)	
40032	TTA Word		40077	TTA Word		40122	Status Bit Field (Bits 15-0)	
40033	TDS Word		40078	TDS Word		40123	Status Bit Field (Bits 31-16)	
40034	TDD Word		40079	TDD Word		40124	Status Bit Field (Bits 47-32)	
40035	TDO Word		40080	TDO Word		40125	Status Bit Field (Bits 63-48)	
40036	Na_2SO_4 Word		40081	Na_2SO_4 Word		40126	DCS Control Bit Field (Bits 15-0)	
40037	Na_2SO_3 Word		40082	Na_2SO_3 Word		40127	LCD Control Bit Field (Bits 15-0)	
40038	Na_2S_2O3 Word		40083	$Na_2S_2O_3$ Word				
40039	Cl^- Word		40084	Cl^- Word				
40040	Sample Temperature Word		40085	Sample Temperature Word				
40041	Reserved Word		40086	Reserved Word				
40042	Reserved Word		40087	Reserved Word				
40043	Reserved Word		40088	Reserved Word				
40044	Reserved Word		40089	Reserved Word				

Notes:

1. Offset is assumed to be ZERO but can be set to any value.
2. Function Code 3 & Function Code 16 are the only codes that are recognized.
3. Function Code 16 can only access register 126.
4. LCD Control Bit Field (Register 127) is a read-only register
5. Floating Point numbers are encoded in an *n*-bit integer where *n* is normally 16 but can be changed to a lower resolution if desired
6. Engineering units can be changed to whatever is desired through the LCD touch panel interface

Ranges For Floating Point Numbers					
Parameter	**Low Val.**	**Hi Val.**	**Units**	**Low Int.**	**Hi Int.**
EA	0	200	lb/100gal	0	$2^n - 1$
AA	0	200	lb/100gal	0	$2^n - 1$
TTA	0	200	lb/100gal	0	$2^n - 1$
Na_2SO_4	0	200	lb/100gal	0	$2^n - 1$
TDS	0	100	%	0	$2^n - 1$
TDD	0	100	%	0	$2^n - 1$
TDO	0	100	%	0	$2^n - 1$

Appendix A.12 Troubleshooting

Table A.12-1. Troubleshooting Guide.

PROBLEM	POSSIBLE CAUSE	SOLUTION
Analyzer will not power on	Power cord not plugged in securely	Check insertion of power cord at analyzer and outlet
	Power is not supplied to outlet	Verify power is being supplied to outlet
	Blown fuse	Check fuse (fuse holder located next to analyzer power switch), if blown replace (3.5 A)
	Internal analyzer temperature too high (triggering thermal shut down switch)	Check internal cooling fan
		Relocate analyzer to a location with a cooler ambient temperature
		Repower once the analyzer has cooled down
	Other	Contact R. E. Hodges, LLC
Printer not working	Power cord not plugged in securely	Check insertion of power cords at outlet, power unit, and printer
	Interface cable not connected properly or securely	Check interface cable connection
	Interface cable connection pins have developed a layer of corrosion	Clean connection pins on interface cable
	Out of paper	Replace paper roll (Appendix A.9)
	Printer needs cleaning	Clean printer (Appendix A.9)
	Other	Contact R. E. Hodges, LLC
LCD panel does not power on or display correctly	LCD panel connection cable not plugged in properly	Disconnect and reconnect LCD panel cable
	LCD cable connection pins have developed a layer of corrosion	Clean connection pins of cable
	Analyzer needs to "REBOOT"	Switch analyzer power OFF, wait 15 seconds, switch power ON
	Other	Contact R. E. Hodges, LLC
Difficult to draw sample into cuvette	Flow restriction	Inspect tubing and cuvette for obstruction or buildup, clean or replace if necessary
		Inspect pipetting bulb, clean or replace if necessary
	Other	Contact R. E. Hodges, LLC
Measurements become erratic or unstable	Cuvette needs to be acid cleaned	Acid clean cuvette (Appendix A.5)
	Sample needs to settle longer in cuvette	Adjust scan parameters (Sec. 4.4.3)
	Light source (bulb or connection) may be loose	Ensure bulb is secure in holder and wire connection is secure and making good contact (Caution: light source and holder will be HOT)
	Light source may be nearing end of lifespan	Check bulb intensity and replace if necessary (Appendix A.10)
	Other	Contact R. E. Hodges, LLC

Table A.12-1 is a troubleshooting guide for some problems that are easily remedied. Other problems could arise that will likely be hardware related and require servicing by R. E. Hodges, LLC.

Appendix A.13 Replacement Parts

Table A.13-1. Replacement Parts List.

PART	DESCRIPTION	REH PART NO.
Analyzer Power Cord	AC, 6 ft	LLA-001
Enclosure Key	Key for opening/closing SS analyzer cabinet	LLA-002
LCD Control Panel	Touch screen user interface	LLA-003
MODBUS Com. Device	MODBUS TCP → MODBUS RTU	LLA-004
NIR Light Source	QTH NIR reflectorized bulb	LLA-005
Pipetting Bulb	60 mL, rubber	LLA-006
Printer	Thermal printer (includes printer power unit)	LLA-007
Printer Cable	Serial interface cable	LLA-008
Printer Paper	Thermal paper, 80 mm width	LLA-009
Printer Power Unit	Power unit, AC power cord, DC power cord	LLA-010
Sample Cuvette	1 mm path length, fused silica	LLA-011
Silicon Tubing	6 mm ID, 4 ft	LLA-012

Appendix A.14 Warranty and Notices

Warranty

The ***DURALYZER-NIR™*** laboratory liquor analyzer is warranted by R. E. Hodges, LLC against defects in material and construction for one year from the date of shipment. Our liability shall be limited to parts repair or replacement. For warranty repair or replacement, the customer shall pay for shipping charges to our facility or travel charges if warranty work is performed at the customer's plant or facility. This warranty does not cover damages or defects caused by improper use, neglect, accidents, wear, inadequate maintenance, poor site preparation, or modifications and repairs not authorized by R. E. Hodges, LLC or explicitly described in this manual. R. E. Hodges, LLC will assume no liability for any incidental, indirect, or consequential damages arising out of the use or misuse of this product.

Notices

- Information contained in this manual is subject to change without notice.
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- Service/maintenance agreements are available, contact R. E. Hodges, LLC for details and pricing.
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